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ISENTROPIC BULK MODULUS: DEVELOPMENT OF A FEDERAL TEST METHOD

INTERIM REPORT
TFLRF No. 465

by
Scott A. Hutzler

U.S. Army TARDEC Fuels and Lubricants Research Facility
Southwest Research Institute® (SwRI®)
San Antonio, TX

for
Mr. Eric Sattler
U.S. Army TARDEC
Force Projection Technologies
Warren, Michigan

Contract No. W56HZV-09-C-0100 (WD19 and WD21-Task 2.1)

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Gary Bessee

Gary B. Bessee, Director
U.S. Army TARDEC Fuels and Lubricants
Research Facility (SwRI®)

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14. ABSTRACT

The overall objective of this effort was to construct additional bulk modulus units based on the previous prototype built at Southwest Research Institute (SwRI). The current methodology allows the measurement of isentropic bulk modulus via speed-of-sound and density at temperatures ranging from 30-80 °C and applied pressures of 1,000-18,000 psi. This method has been applied successfully to aviation turbine fuels and diesel fuels composed of petroleum, synthetic, and alternative feedstocks and is currently a requirement in fit-for-purpose (FFP) testing toward qualification of aviation fuels and additives. A round robin test program was carried out on selected fuels and precision data calculated. A Federal Test Method (FTM) was drafted based on the current methodology and supporting round robin data. The results from this effort have demonstrated that these units have the ability to provide accurate and repeatable data across a range of temperatures and pressures.

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EXECUTIVE SUMMARY

The overall objective of this effort was to construct additional bulk modulus units based on the previous prototype built at Southwest Research Institute (SwRI). The current methodology allows the measurement of isentropic bulk modulus via speed-of-sound and density at temperatures ranging from 30-80 °C and applied pressures of 1,000-18,000 psi. This method has been applied successfully to aviation turbine fuels and diesel fuels composed of petroleum, synthetic, and alternative feedstocks. Bulk Modulus, currently referenced as ASTM D6793, is a requirement in fit-for-purpose (FFP) testing toward qualification of aviation fuels and additives (ASTM D4054). The units were delivered to select government labs and training was provided to their personnel. A round robin test program was carried out on selected fuels and precision data calculated. A Federal Test Method (FTM) was drafted based on the current methodology and supporting round robin data.

The results from this effort have demonstrated that these units have the ability to provide accurate and repeatable data across a range of temperatures and pressures. Reproducibility among the labs is largely impacted by the specific calibration on each unit and the variation of conditions. The experience of the operators appears to have had a negligible impact on the results. The overall methodology should have sufficient resolution to differentiate fuel types (e.g., IPK vs. Jet A vs ULSD).

Recommendations for future work include a series of smaller-scale round robin tests among the participating labs with tighter control on test conditions, expanding into higher temperature and pressure regimes, and developing a new design for the high pressure cell in which the acoustic sensor is immersed in the high-pressure environment thus improving the accuracy of the speed-of-sound measurements.

FOREWORD/ACKNOWLEDGMENTS

The U.S. Army TARDEC Fuel and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, performed this work during the period September 2012 through March 2015 under Contract No. W56HZV-09-C-0100. The U.S. Army Tank Automotive RD&E Center, Force Projection Technologies, Warren, Michigan administered the project. Mr. Eric Sattler (RDTA-SIE-ES-FPT) served as the TARDEC contracting officer's technical representative. Nichole K. Hubble of TARDEC served as project technical monitor.

The authors would like to acknowledge the contribution of the TFLRF technical support staff including Keri Petersen and Cody Wyatt along with the administrative and report-processing staff.

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ACRONYMS AND ABBREVIATIONS

ATJ	alcohol-to-jet
FFP	fit-for-purpose
DAQ	Data Acquisition System
RTD	Resistive Temperature Device
g	gram
kg	kilogram
m	meter
Pa	pascal
psi	pounds per square inch
r	repeatability
R	reproducibility
s	second
ULSD	ultra-low sulfur diesel
USB	Universal Serial Bus

1.0 BACKGROUND AND OBJECTIVE

The overall objective of this effort was to construct additional bulk modulus units based on the previous prototype built at Southwest Research Institute (SwRI). The current methodology allows the measurement of isentropic bulk modulus via speed-of-sound and density at temperatures ranging from 30-80 °C and applied pressures of 1,000-18,000 psi. This method has been applied successfully to aviation turbine fuels and diesel fuels composed of petroleum, synthetic, and alternative feedstocks. Bulk Modulus, currently referenced as ASTM D6793, is a requirement in fit-for-purpose (FFP) testing toward qualification of aviation fuels and additives (ASTM D4054). Once constructed, the units were to be delivered to selected government labs and training provided to their personnel. A round robin test program would then be carried out on selected fuels and precision data calculated. The precision measurements would be used to support the creation of a Federal Test Method (FTM).

2.0 APPARATUS

2.1 HARDWARE

The current version of the isentropic bulk modulus apparatus (shown in Figure 1) is an assembly of the following components. The primary upgrade from the previous version was the addition of a process densitometer and display to provide more accurate density measurements.

- Oven – range of 30-100 °C, stable within 1 °C
- Data Acquisition System (DAQ) – for recording pressure, volume, temperature, and density data
- Bulk Modulus Excel Toolkit
- Digital Oscilloscope – for measuring pulse echoes
- High Pressure Generator (rated for 60K psi)
- Flow loop and valving (rated for 60K psi)
- High Pressure Cell (rated for 30K psi max)
- Pressure Transducer (50K psi)
- Pulser/Receiver Module – to drive the acoustic transducer

- Acoustic transducer
- Process Densitometer (rated for 20K psi and 200 °C)
- Densitometer Control/Display
- Resistive Temperature Device (RTD)
- Two (2) 500 mL beakers or sample bottles for sampling and waste
- Secondary containment for beakers/sample bottles
- Peristaltic pump and fuel compatible hose – for pumping fluid into the system

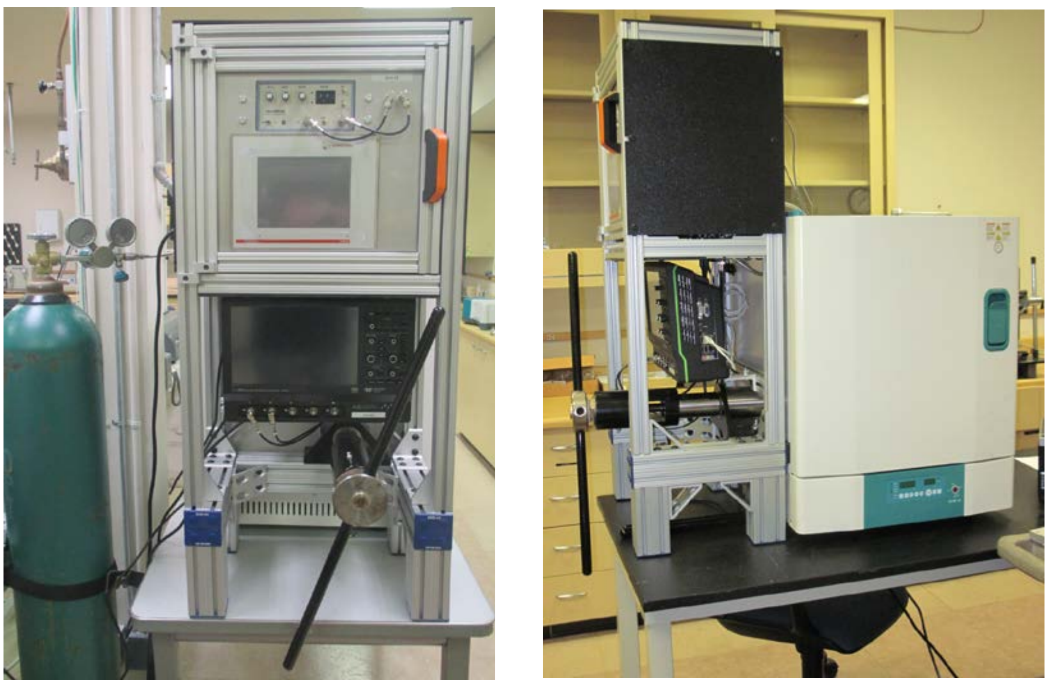


Figure 1. Bulk Modulus Apparatus – Exterior

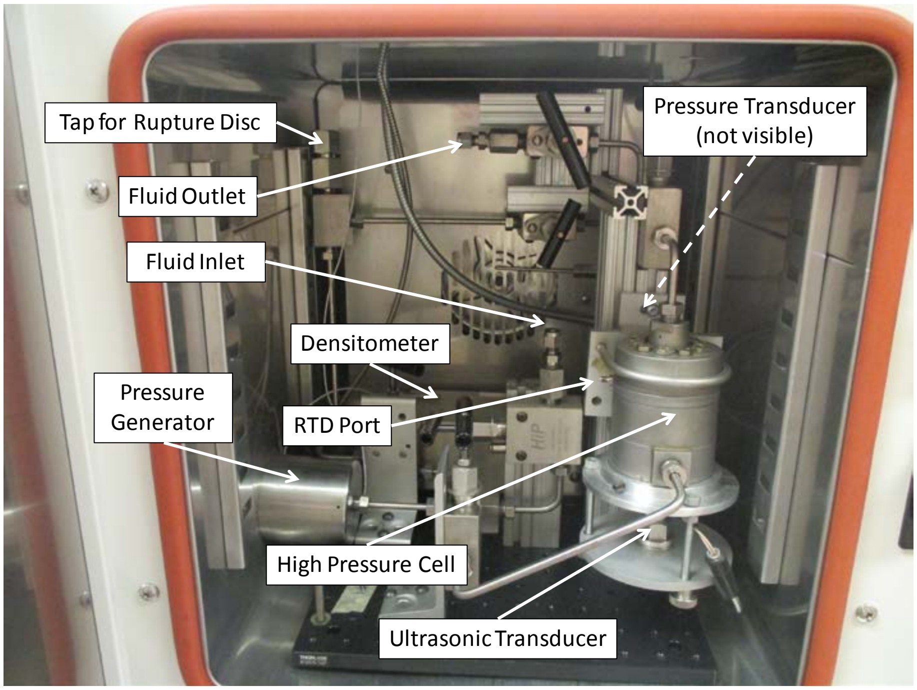


Figure 2. Bulk Modulus Apparatus – Interior

2.2 SOFTWARE

Although software development was never envisioned for this effort, it became quite clear that some type of data management was needed. To aid the operator in calibration/validation, and collecting and analyzing data, a Bulk Modulus Excel Toolkit (Figure 3) was developed. This toolkit provides a number of useful tools, in addition to, some built in algorithms that can compute the expected values of speed-of-sound and density for water and n-pentane as a function of temperature and pressure. This toolkit can also interact with an onboard USB-DAQ system to stream live data into Excel which makes data collection more efficient and provides access to historical data for an entire run.

Further details concerning the use and features of the Bulk Modulus Excel Toolkit can be found in the Operator's Manual.

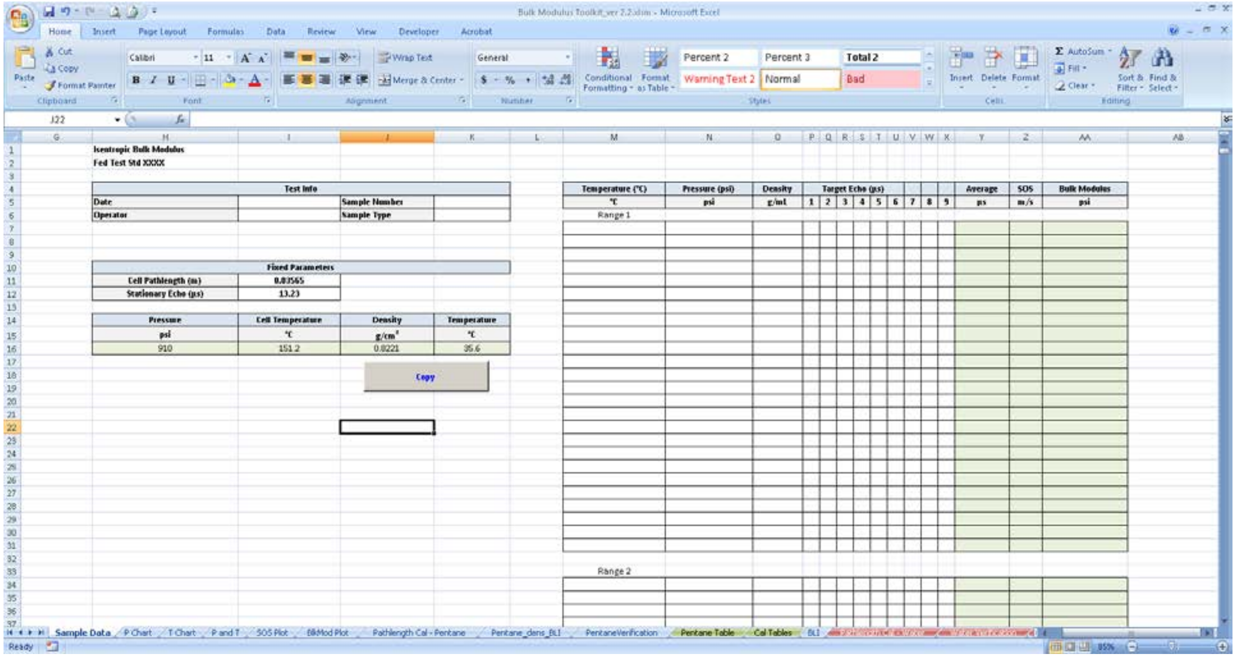


Figure 3. Bulk Modulus Excel Toolkit

3.0 ROUND ROBIN TESTING

3.1 ROUND ROBIN PARTICIPANTS

The four labs that participated in the round robin study included:

- TARDEC Fuels & Lubricants Research Facility (TFLRF) at Southwest Research Institute (SwRI)
- TARDEC Fuels Laboratory – Detroit Arsenal
- Naval Air Station Patuxent River (NAVAIR)
- Air Force Research Laboratory (AFRL) – Wright Patterson AFB

Personnel from SwRI traveled to each destination, set up and configured each instrument, and provided training to on-site personnel. The 5th unit was scheduled to be delivered to the National Renewable Energy Laboratory (NREL) but NREL was forced to withdraw from the round robin due to safety concerns. So the 5th unit was set back up at TFLRF/SwRI and the round robin was conducted using a newly trained operator to add variability. The 5th unit was eventually installed at Michigan State University under the direction of Dr. Carl Lira.

3.2 ROUND ROBIN PROTOCOL

3.2.1 Test Fuels

For the round robin testing, five fuels were chosen that would span a large region of the bulk modulus space from synthetic iso-paraffinic fuels to petroleum-based diesel fuels. The test fuels are identified in Table 1. A testing order and file naming convention was provided as shown in Table 2.

Table 1. Test Fuels

Sample ID	Fuel Type
6065	F-76
6484	50/50 ATJ/Jet A
6485	ULSD
6486	ATJ
6487	Jet A

Table 2. Test Order

Test Order	Filename
1. 6485	{TARDEC, AFRL, NAVAIR}_35C_6485
2. 6485	{TARDEC, AFRL, NAVAIR}_65C_6485
3. 6486	{TARDEC, AFRL, NAVAIR}_35C_6486
4. 6486	{TARDEC, AFRL, NAVAIR}_65C_6486
5. 6065	{TARDEC, AFRL, NAVAIR}_35C_6065
6. 6065	{TARDEC, AFRL, NAVAIR}_65C_6065
7. 6484	{TARDEC, AFRL, NAVAIR}_35C_6484
8. 6484	{TARDEC, AFRL, NAVAIR}_65C_6484
9. 6487	{TARDEC, AFRL, NAVAIR}_35C_6487
10. 6487	{TARDEC, AFRL, NAVAIR}_65C_6487

3.2.2 Validation Sample

Prior to running actual round robin samples, each participating lab was asked to run a sample of n-pentane to verify proper operation of the unit and to validate the operator’s ability to collect data properly.

The following notes were provided for the pentane run:

- Run the n-pentane at 35 °C
- Measuring at a specific pressure is not critical in this step. This is more about validating the instrument against a standard for which the speed-of-sound and density values are already established.

- Try to measure about eight (8) different pressures between 1,000-12,000 psi. The goal is to find the best and strongest signals in that range. Hitting an exact pressure is not critical. The key element to this test is finding a clear strong signal.
- Use a copy of the provided Excel toolkit to record temperature, pressure, density, and all the target echoes.

3.2.3 Test Conditions

The target temperatures and pressures for the actual round robin samples are shown in Table 3 and Table 4, respectively. In each table, a tolerance for the target condition is provided. For the temperature set point, a tolerance of ±1 °C was provided to accommodate slight differences in each oven and the ability of the ovens to remain stable at a given temperature for long periods of time. The tolerance in test pressure was provided to allow some flexibility in obtaining a clean signal on the digital oscilloscope.

Table 3. Test Temperature (with allowable tolerance)

Temperature	+/-
35 °C	1
65 °C	1

Table 4. Test Pressures (with allowable tolerance)

Pressure (psi)	+/-
1000	50
2000	100
3000	100
4000	100
5000	100
6000	100
7000	100
8000	100
9000	100
10000	100
11000	100
12000	100
13000	100
14000	100
15000	100

3.3 ROUND ROBIN PROCEDURE

The following notes were provided to the participating labs. The reader may reference APPENDIX G for a more detailed description of unit operation.

Test Notes:

- Testing is to be performed in the same manner as was accomplished with the n-pentane with a few exceptions.
- It might be advisable to check the grease between the acoustic transducer and the bottom of the cell if you haven't refreshed it in the last week. It will improve the signal strength.
- Use the filename indicated above with the appropriate {Lab} substituted at the beginning
- Temperature and Pressure tolerances are noted in the tables above. These are important so we receive data as near to those as possible for comparison.
- You may need to set the oven one degree higher or lower to get closer to the expected test temperature.
- Use the temperature indicated on the *main display panel* to monitor temperature. Wait until the temperature on the main display is stable to within a few tenths of a °C over a ten minute period before taking measurements. However, also monitor the cell temperature recorded through the DAQ. Make sure that the two temperatures are within 1-2 °C and stable.
- The pressure tolerance is provided to allow you some flexibility at each pressure point to find the best possible signal. In some cases, some of you may not be able to see a valid signal at all (it might disappear or get lost in the noise). In that case, skip that point and move on.
- Once you've reached the desired pressure, allow the system to settle for about a minute. Make sure that the pressure curve seen on the main display has flattened out and is not dropping.
- Record all data into the provided Excel toolkit using the "Copy" button on the first tab.

Flushing

- In between samples, use 200-300 mL of the next sample to flush the system and discard the fluid. Operate the system as if you were filling it to test. Force sample through both legs of the system and cycle the pressure generator in and out several times to ensure it gets flushed also.
- Replace the test fluid with a new 200-300 mL batch.
- Using about half of this fresh sample, flush the system again by pushing fluid through both legs and cycle the generator once. Direct this fluid to a waste container.
- Switch to recirculation mode by directing the exiting fluid back to the same sample container from which it's being drawn.
- At this point, make sure to apply back pressure to the exit hose and keep circulating to eliminate air from the system as you did with the n-pentane.

Test Sequence

- In the interest of time, it was agreed that the three replicate runs at each temperature would be accomplished without reloading of the system.
- Make note of the starting pressure after all the valves in the system have been closed but before raising the pressure. It will not be zero but you will reference this point as a baseline (approx 0-200 psi typically).
- Once you've cycled through all of the pressures, return the pressure to the baseline noted above and repeat the testing.
- It's unlikely that both temperatures can be completed in one day. The best approach might be to lower the pressure to the baseline reference point and turn off the oven. The next day, repack the system using the same fluid and run the higher temperature. Remember: NEVER RAISE THE TEMPERATURE OF THE OVEN UNLESS THE PRESSURE IS AT OR NEAR ATMOSPHERIC. The pressure will increase as the temperature rises. Once at temperature, you can back off the pressure generator to return to a lower pressure to make the first measurements. This is another reason to only load the pressure generator about 2/3 full (stem extended about 2/3 out) so you have some allowable travel in the opposite direction.

3.4 ANALYSIS OF ROUND ROBIN DATA

Precision data in the form of repeatability (r) and reproducibility (R) for the round robin testing was generated according to ASTM E691 – *Conducting an Interlaboratory Study to Determine the Precision of a Test Method*. For the time-being, this method was chosen over the more common method ASTM D6300 - *Standard Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants*. E691 fits better with the selected sample matrix and number of replicates in this study and is more flexible in terms of rejecting data that was clearly caused by an operator error.

4.0 RESULTS AND DISCUSSION

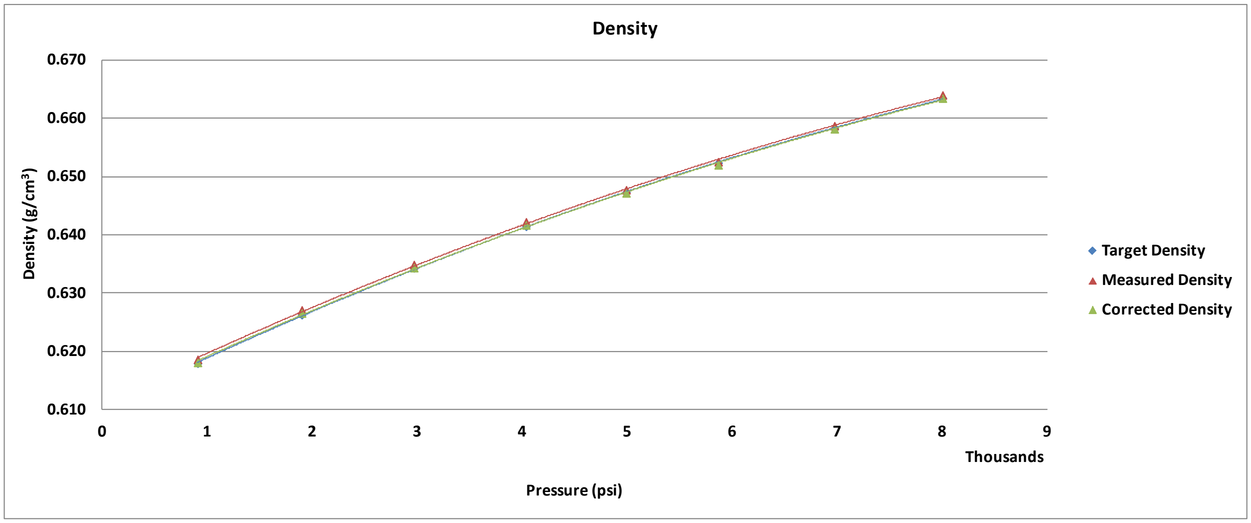
4.1 POST-TEST ANALYSIS OF PENTANE RESULTS

Following the delivery of the final unit, a new feature was added to the software that allows the user to map density and speed-of-sound data to known values for n-pentane. Prior to this, validation was being performed point-by-point in a semi-quantitative manner and only on a limited number of points. The new feature allows a full curve for both measurements to be plotted for any number of pressure points at a fixed temperature. Sections 4.1.1 - 4.1.5 below, show what happens when the raw n-pentane data from each lab is entered into the software. In a few cases, there were some substantial deviations from the target value. These can be easily corrected in the system prior to running actual samples or even post analysis.

This data is probably the most telling of all and confirms that the measured values generally follow the correct trends but simply require an offset to bring them in-line with established values.

The precision data reported herein have not undergone any substantial corrections.

4.1.1 n-Pentane Data, Lab A



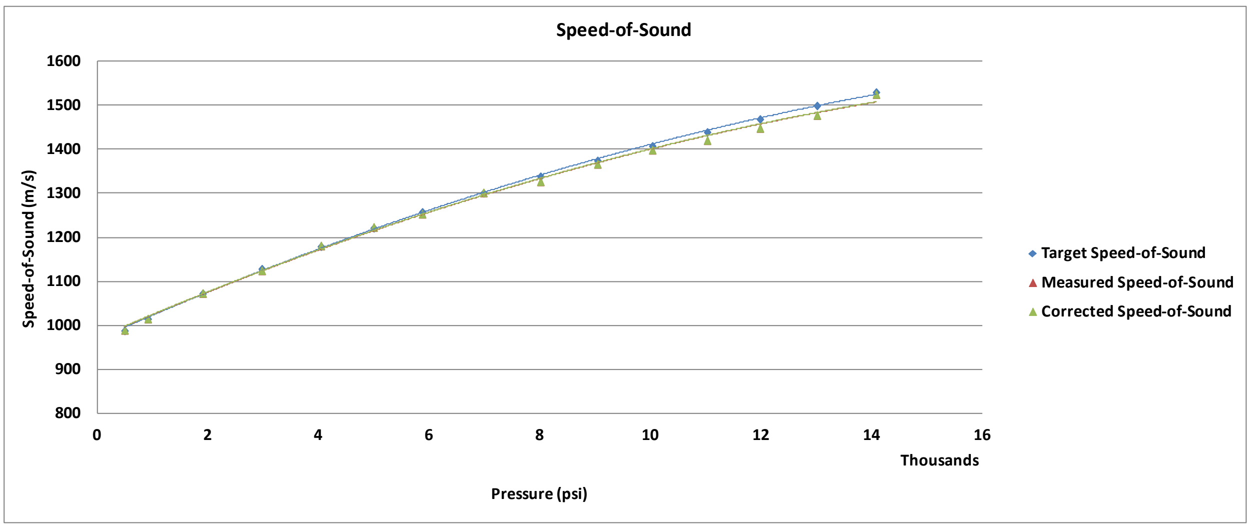
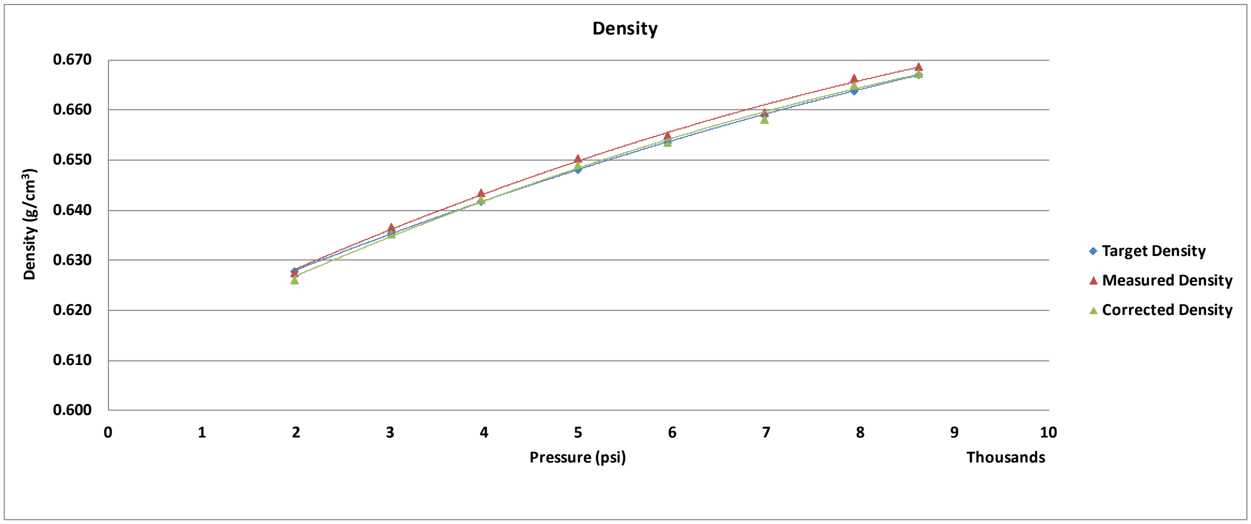


Figure 4. Lab A, Validation using n-Pentane

4.1.2 n-Pentane Data, Lab B



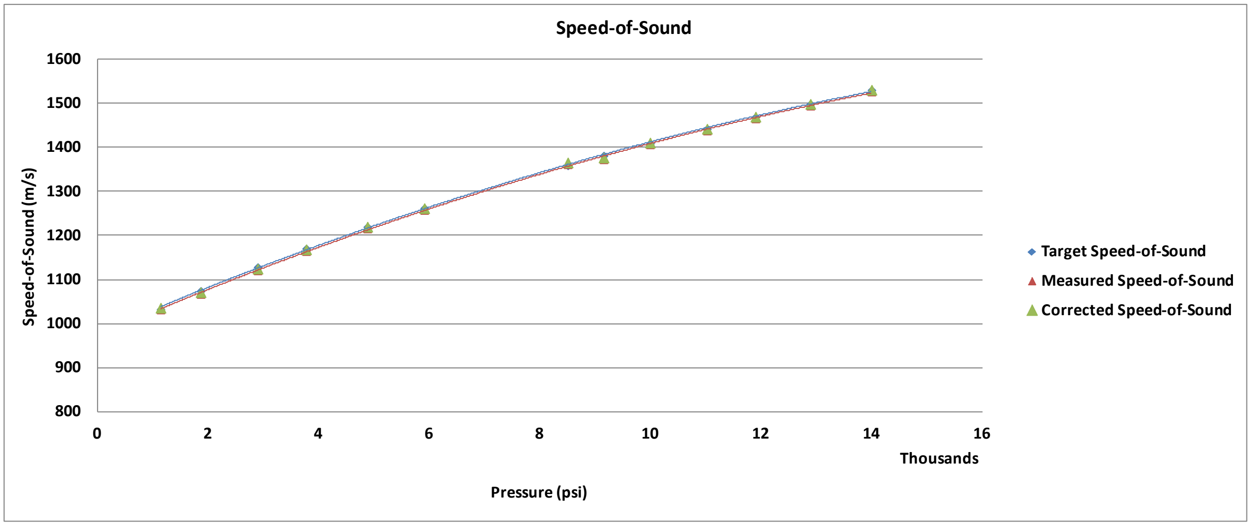
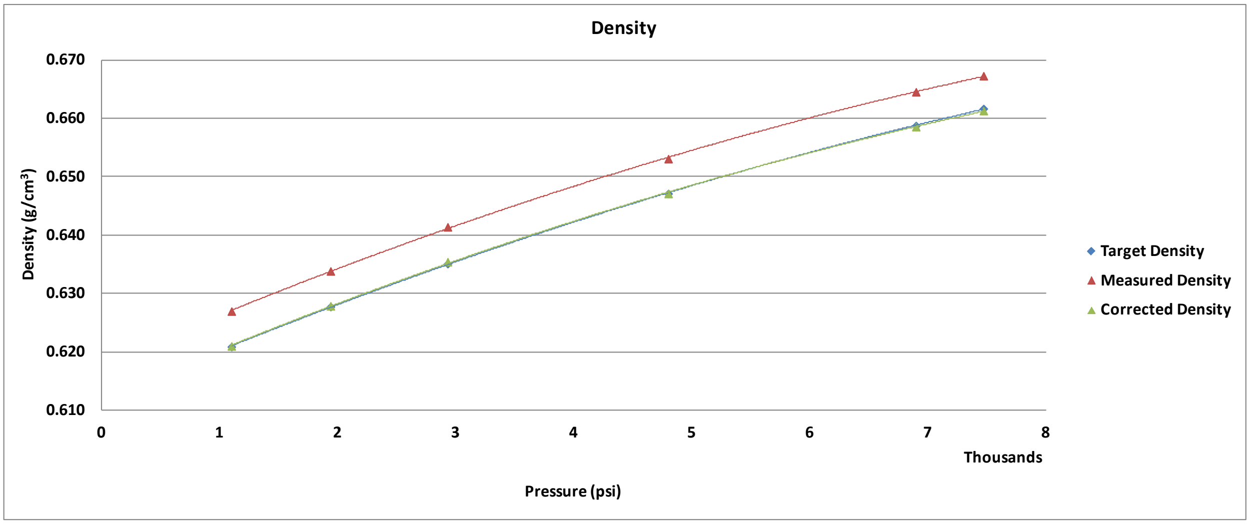


Figure 5. Lab B, Validation using n-Pentane

4.1.3 n-Pentane Data, Lab C



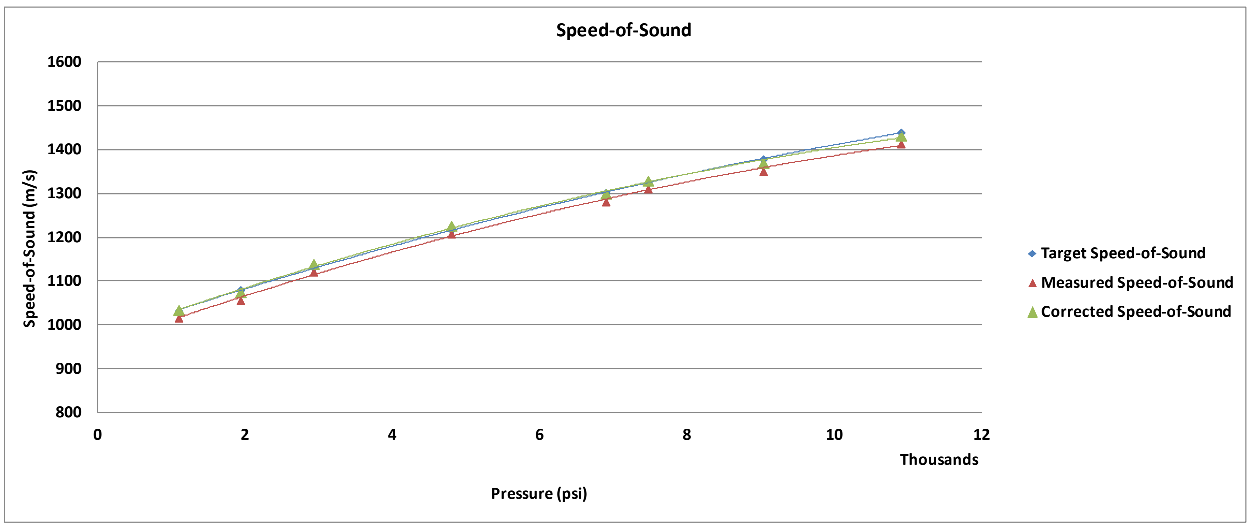
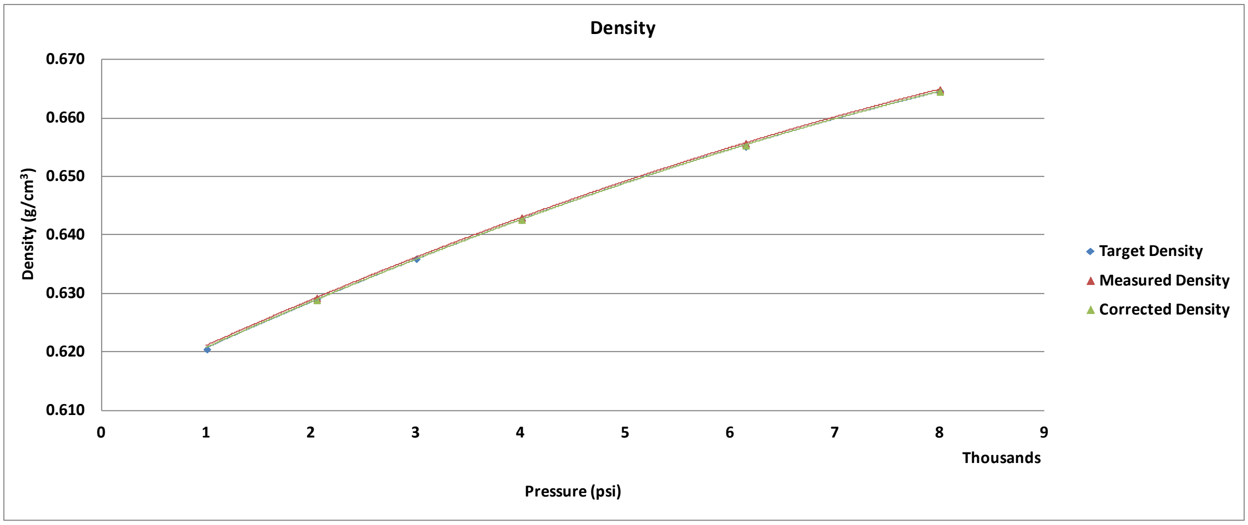


Figure 6. Lab C, Validation using n-Pentane

4.1.4 n-Pentane Data, Lab D



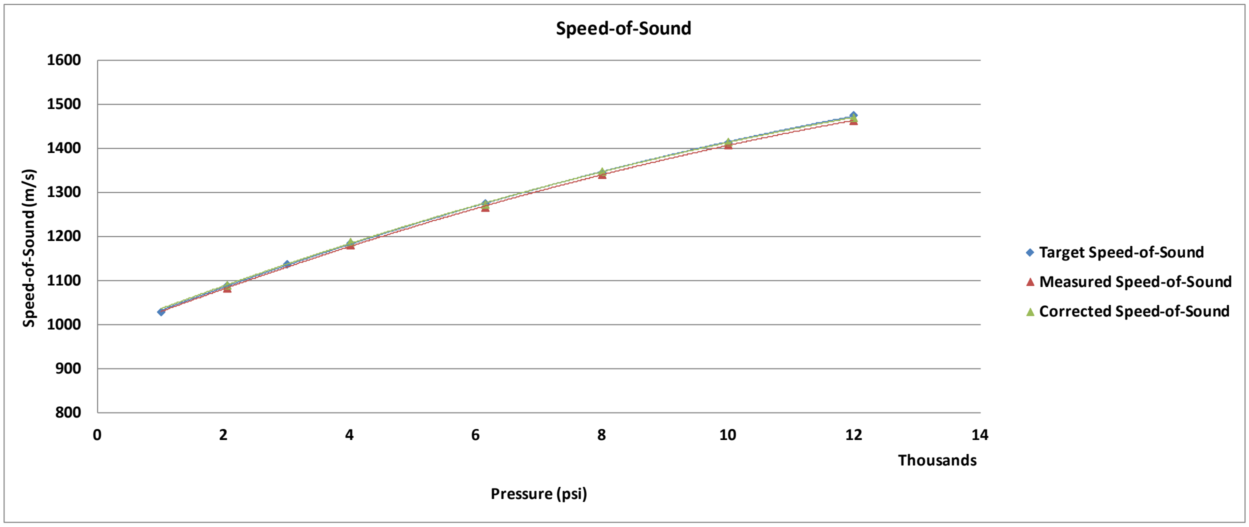
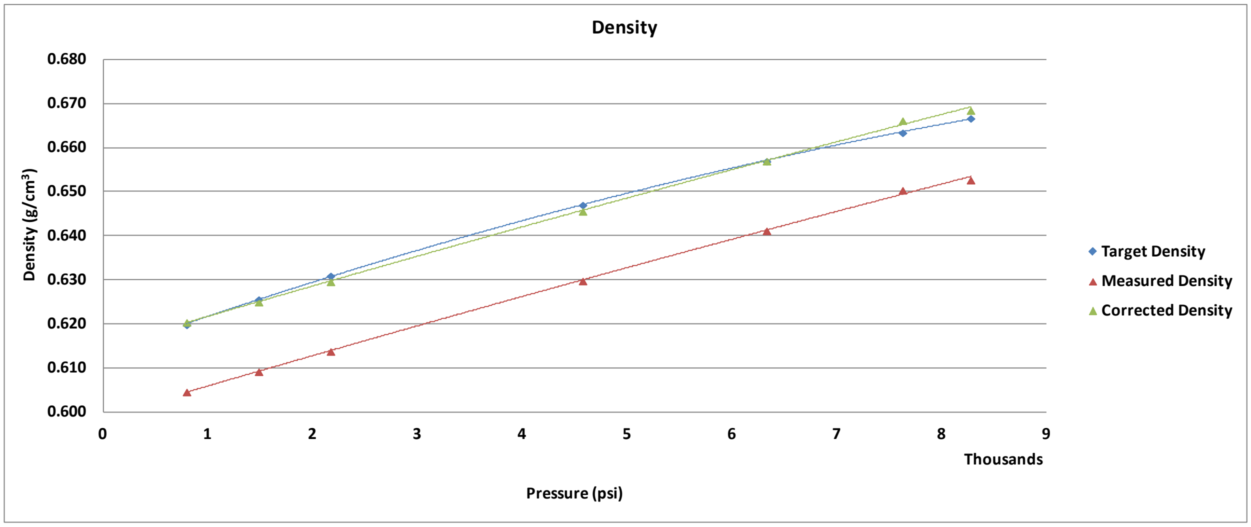


Figure 7. Lab D, Validation using n-Pentane

4.1.5 n-Pentane Data, Lab E



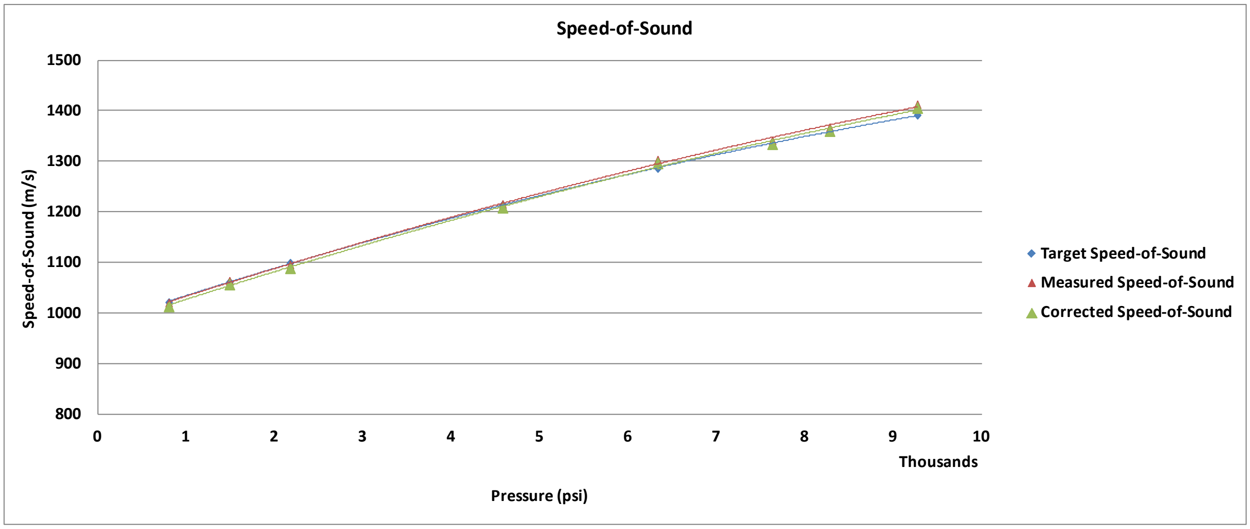


Figure 8. Lab E, Validation using n-Pentane

4.2 NOMINAL RESULTS (LAB A)

Figure 9, Figure 10, and Figure 11 show data that is representative of the overall technique and the type of performance that should be expected from each system. As expected, the speed-of-sound and bulk modulus curves are similar for the ULSD and F-76 fuels, and the ATJ/Jet A blend falls somewhere between its two blendstock components.

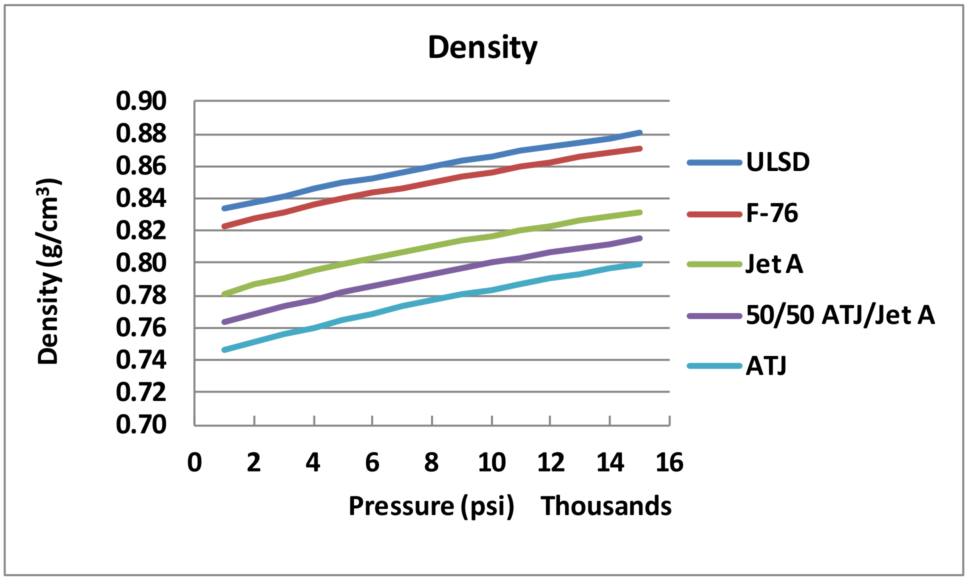


Figure 9. Representative Density Data for the Test Fuels @ 35 °C

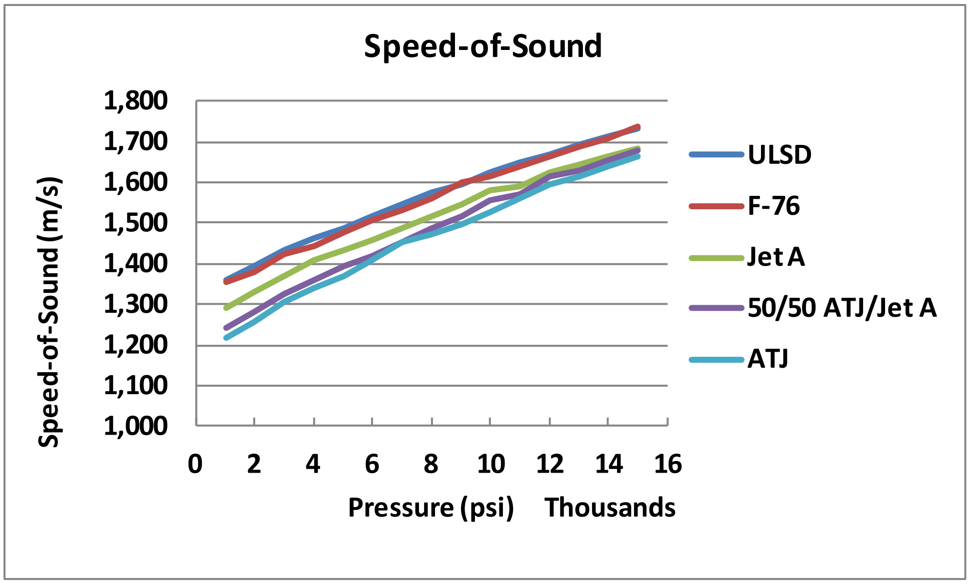


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Figure 10. Representative Speed-of-Sound Data for the Test Fuels @ 35 °C

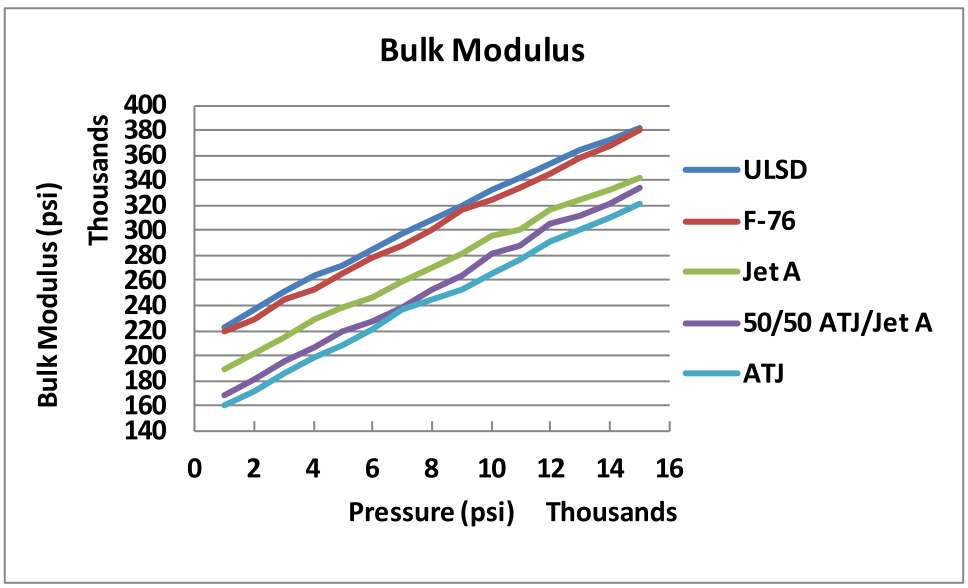


Figure 11. Representative Bulk Modulus Data for the Test Fuels @ 35 °C

4.3 PRECISION DATA

The final r and R values for each of the fuels is tabulated in APPENDIX A. Each of the fuels is presented in numerical order and consists of a table of precision data at each pressure point and a graphical representation of the same for each measurement at a fixed temperature. It's clear to see that the repeatability on a given unit is generally consistent across the pressure range and the values are not unreasonable. The reproducibility shows a fairly consistent trend across all samples that indicates increasing error at higher pressures. The source of this is still being debated since that trend is not evident in the repeatability.

4.4 TEMPERATURE COMPARISON

Plots of comparative temperature data are provided in Appendices B-F. This shows that, for a given fuel on a select unit, the shapes of the temperature curves are very consistent. However, the separation of the curves does vary somewhat between labs. Some of the lab data appears more noisy than others. This is more than likely an operator-induced noise caused by inconsistent measurement technique on the oscilloscope.

5.0 FEDERAL TEST METHOD (FTM)

Based on the procedures developed under this effort, a draft federal test method (FTM) was written for this apparatus. Several existing test methods were reviewed to understand the typical style and manner in which the federal test methods are written. A copy of the draft FTM in included in APPENDIX G.

6.0 CONCLUSIONS

The precision data collected under this effort and the collective experience of working with five different units leads to the following tentative conclusions:

- One or two of the labs appears to be skewing the reproducibility the most. This is a direct result of less than optimum calibration adjustment prior to testing. The overall results would likely improve if a post-test calibration adjustment were performed.
- There appears to be a trend in the reproducibility data as a function of pressure. Since that variation is not seen in the repeatability data, it’s difficult to conclude that the problem is systematic. This will need to be researched further.
- Each unit has the propensity to be accurate and repeatable across a range of temperatures and pressures.
- The accuracy on any given unit is solely based on the individual calibrations for pressure, temperature, density, and speed-of-sound. The allowable tolerances in pressure in this round robin test were likely too wide, causing larger than expected differences between the labs. The next set of testing should focus on specific sets of conditions to eliminate that variation. While temperature is important, the ovens currently being used seem adequately stable provided they are accurate.
- Reproducibility among the labs is largely impacted by the specific calibration on each unit and the variation of conditions. To a lesser extent, the experience of the operators can impact the results but, in general, it appears that most operators became relatively proficient in a short time.
- The density calibration appears to be holding up well over time with little or no change in slope and might only require periodic adjustments to account for drift. These adjustments

can be determined by running a standard, such as n-pentane. New features are being added to the Bulk Modulus Excel Toolkit to automate this adjustment and improve the calibration for both density and speed-of-sound.

- The overall methodology should have sufficient resolution to differentiate fuel types (e.g., IPK vs. Jet A vs ULSD). The only issue seen to date is difficulty in resolving tightly spaced serial dilutions (e.g., 25 vol% vs. 35 vol%).

7.0 RECOMMENDATIONS

Based on the findings in this first round of testing, it may be advisable to conduct a series of smaller-scale, round robin tests among the participating labs. The frequency of testing could be semi-annual. From the experience gained here, one sample run at a single temperature and 10-15 pressure points should be adequate to determine how well the units are operating over time. This could be run similar to an ASTM crosscheck program. The testing could be based on a standard material, like n-pentane. Each lab could acquire their own sample of a specified grade and from a specific source and report the data to a central location for processing.

Ultimately, the desire is to operate at much higher pressures and temperatures. While it may be possible to make adjustments to the current design to achieve those goals, it might be more beneficial to study an entirely new design for the high pressure cell. Once of the limitations of this approach that affects the accuracy is having to shoot the acoustic signal through the cell wall. If a cell and sensor could be developed in which the acoustic sensor is immersed in the high-pressure environment, then speed-of-sound accuracy would likely increase by nearly 1000-fold.

APPENDIX A.

Precision Data Summary

Table A-1. Sample 6065 at 35 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0024**	**0.0015**	**0.0012**	**0.0020**	**0.0009**	**0.0018**	**0.0008**	**0.0007**	**0.0005**	**0.0007**	**0.0002**	**0.0003**	**0.0003**	**0.0017**	**0.0004**	**0.0010**
	R	**0.0169**	**0.0141**	**0.0142**	**0.0137**	**0.0135**	**0.0143**	**0.0150**	**0.0145**	**0.0144**	**0.0145**	**0.0139**	**0.0146**	**0.0148**	**0.0155**	**0.0163**	**0.0147**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**14**	**12**	**2**	**10**	**6**	**12**	**4**	**6**	**7**	**10**	**14**	**5**	**14**	**13**	**3**	**9**
	R	**22**	**27**	**29**	**48**	**48**	**35**	**37**	**49**	**50**	**41**	**54**	**54**	**53**	**51**	**69**	**45**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**4177**	**3781**	**704**	**4009**	**2121**	**4823**	**1733**	**2430**	**2787**	**4223**	**5621**	**2000**	**6011**	**5624**	**1568**	**3441**
	R	**5732**	**8116**	**9996**	**15990**	**15081**	**12699**	**13478**	**18648**	**17994**	**15374**	**21828**	**22426**	**24486**	**23591**	**31499**	**17129**

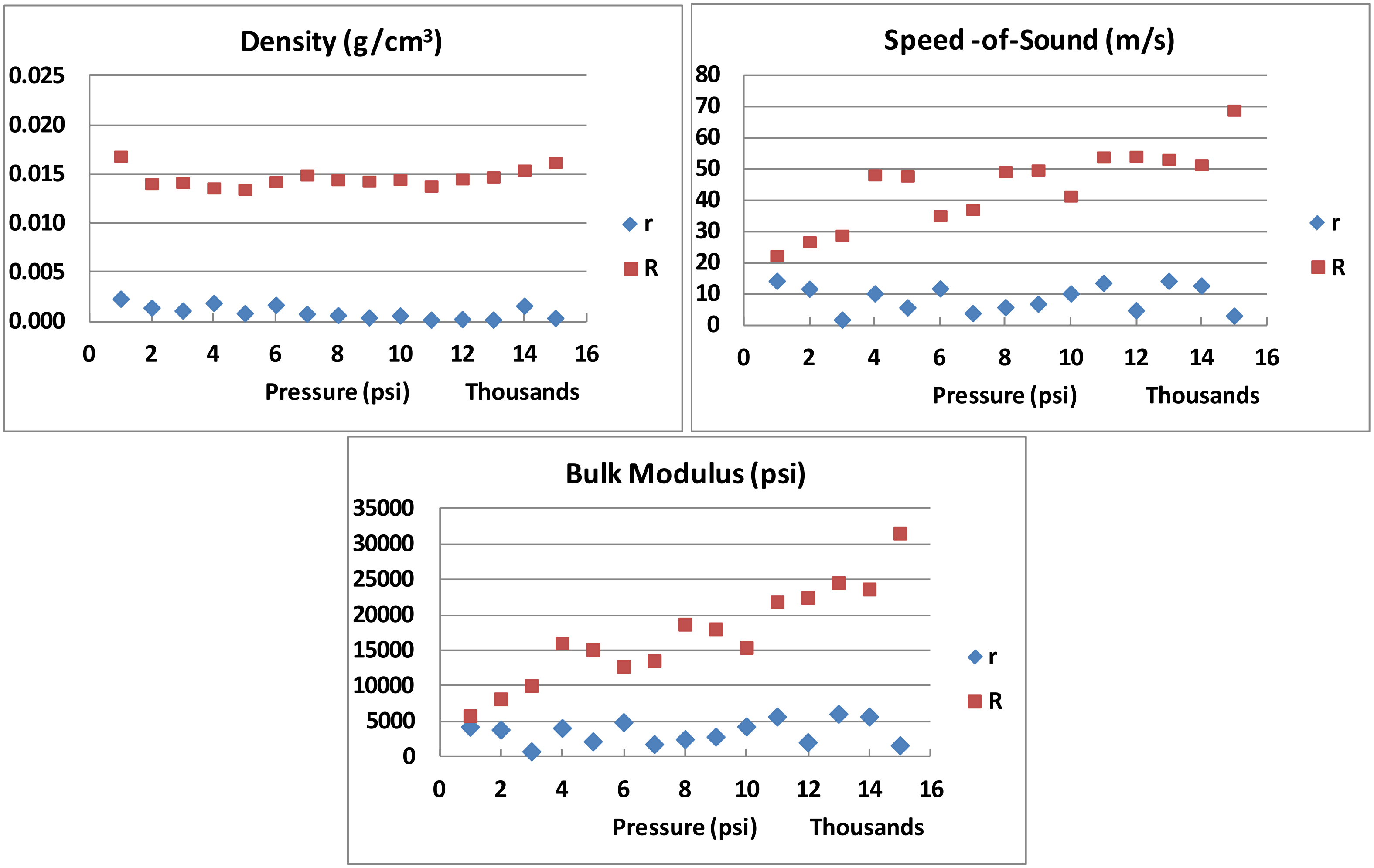


Figure A-1. Sample 6065 at 35 °C

Table A-2. Sample 6065 at 65 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0022**	**0.0024**	**0.0019**	**0.0017**	**0.0025**	**0.0018**	**0.0018**	**0.0020**	**0.0019**	**0.0006**	**0.0020**	**0.0018**	**0.0016**	**0.0017**	**0.0016**	**0.0018**
	R	**0.0201**	**0.0173**	**0.0170**	**0.0171**	**0.0143**	**0.0159**	**0.0181**	**0.0171**	**0.0163**	**0.0178**	**0.0165**	**0.0151**	**0.0161**	**0.0171**	**0.0159**	**0.0168**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**17**	**17**	**2**	**6**	**5**	**13**	**3**	**14**	**7**	**5**	**6**	**9**	**10**	**8**	**3**	**8**
	R	**27**	**22**	**18**	**25**	**20**	**29**	**37**	**37**	**25**	**34**	**35**	**38**	**40**	**55**	**52**	**33**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**4926**	**5354**	**677**	**1628**	**2099**	**4482**	**1570**	**5149**	**2215**	**1909**	**2253**	**3112**	**3737**	**2991**	**1123**	**2882**
	R	**7839**	**7932**	**5193**	**8373**	**7740**	**11488**	**9764**	**13775**	**9949**	**12569**	**12142**	**11778**	**14210**	**21426**	**19151**	**11555**

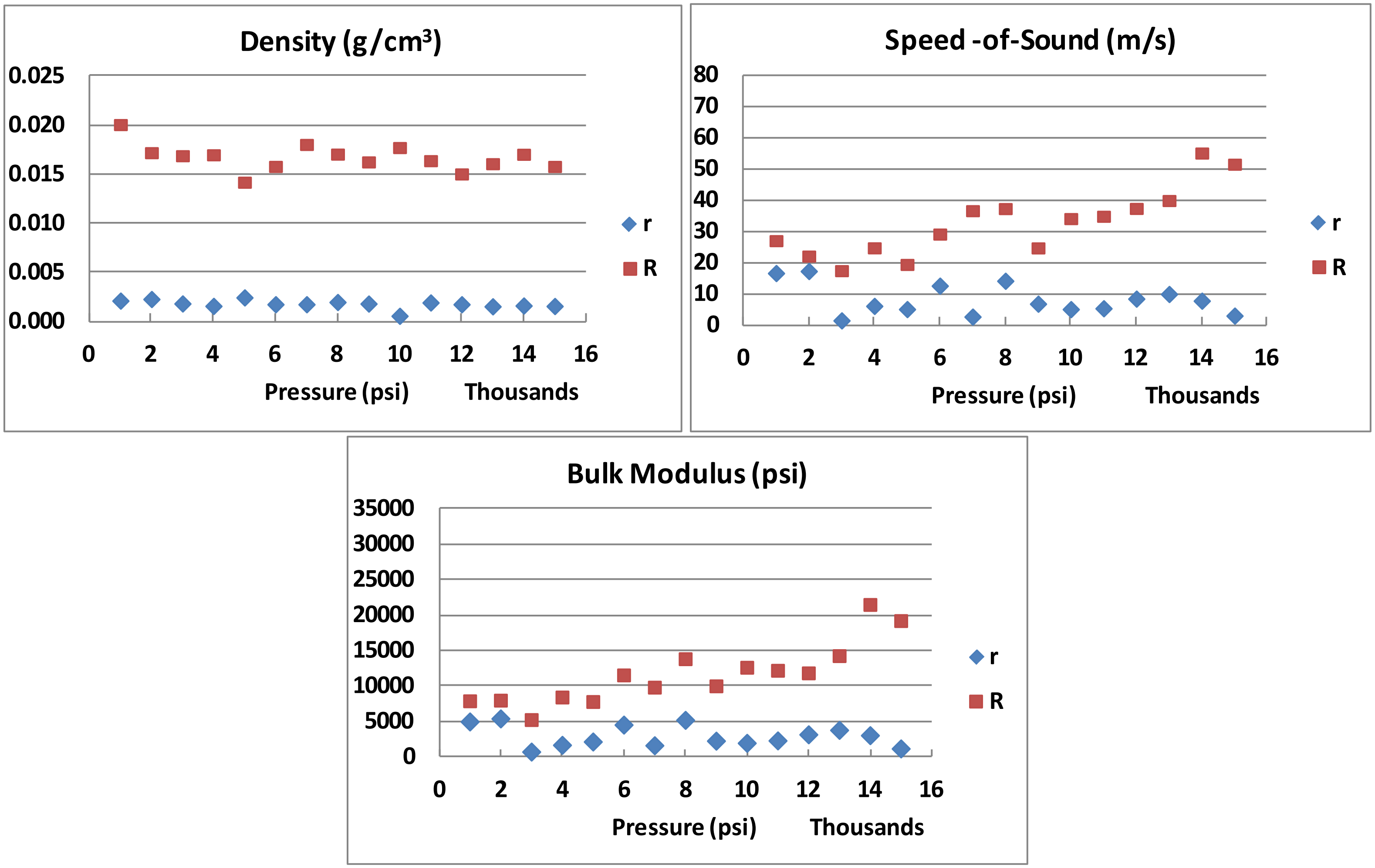


Figure A-2. Sample 6065 at 65 °C

Table A-3. Sample 6484 at 35 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0028**	**0.0027**	**0.0031**	**0.0031**	**0.0043**	**0.0030**	**0.0026**	**0.0023**	**0.0020**	**0.0025**	**0.0027**	**0.0026**	**0.0033**	**0.0031**	**0.0037**	**0.0029**
	R	**0.0108**	**0.0094**	**0.0097**	**0.0114**	**0.0109**	**0.0109**	**0.0101**	**0.0093**	**0.0103**	**0.0102**	**0.0112**	**0.0099**	**0.0106**	**0.0114**	**0.0139**	**0.0107**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**6**	**10**	**15**	**1**	**13**	**6**	**9**	**14**	**2**	**8**	**2**	**12**	**10**	**2**	**4**	**8**
	R	**22**	**37**	**34**	**25**	**38**	**43**	**51**	**50**	**44**	**49**	**56**	**45**	**55**	**64**	**56**	**45**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**1071**	**3016**	**4944**	**854**	**4749**	**1837**	**3297**	**4729**	**696**	**3842**	**1253**	**4316**	**4777**	**941**	**2961**	**2886**
	R	**5105**	**10054**	**9512**	**7357**	**10702**	**13738**	**15035**	**16527**	**14160**	**17300**	**20093**	**16074**	**20658**	**24652**	**27061**	**15202**

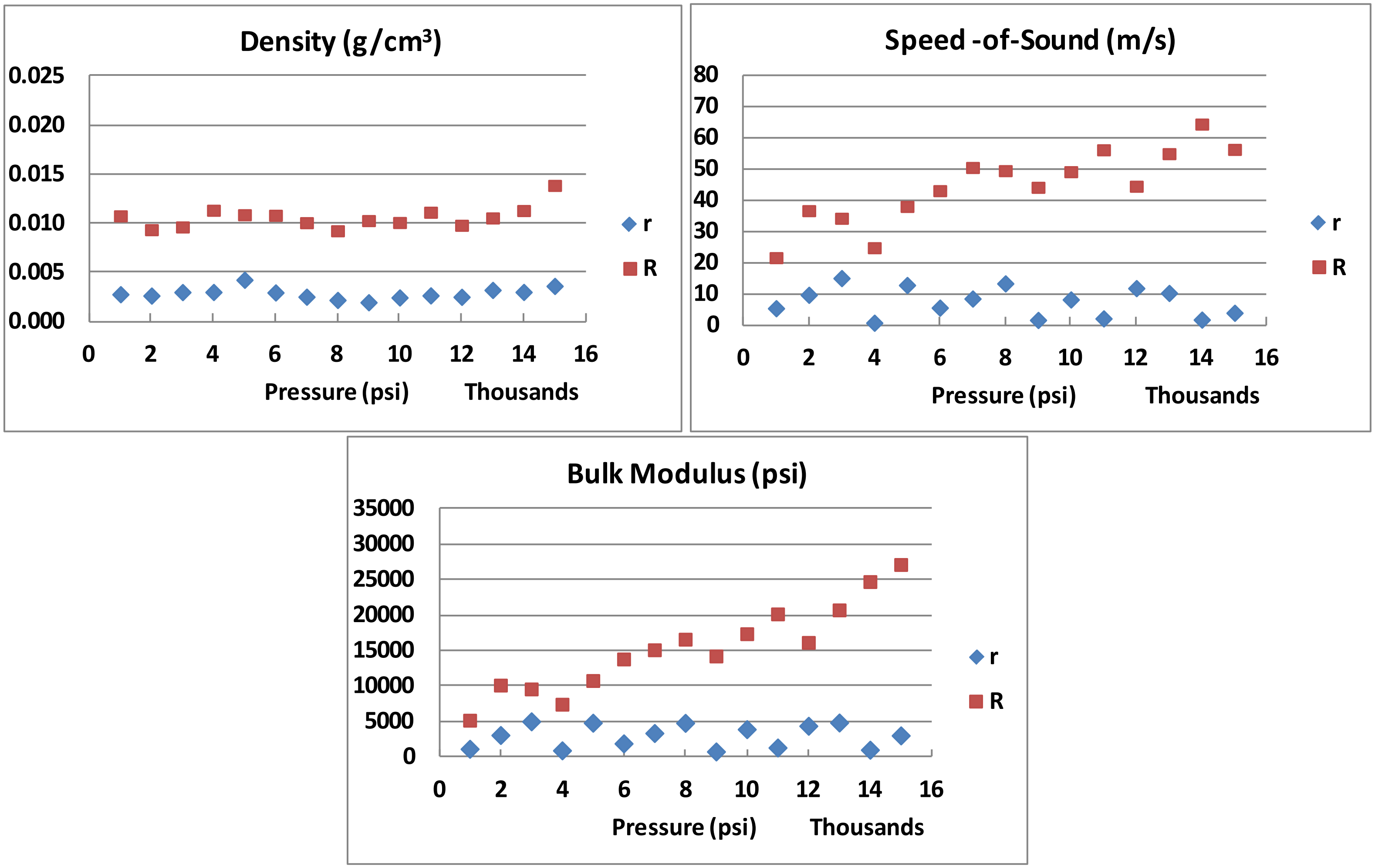


Figure A-3. Sample 6484 at 35 °C

Table A-4. Sample 6484 at 65 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0039**	**0.0019**	**0.0024**	**0.0025**	**0.0017**	**0.0017**	**0.0024**	**0.0017**	**0.0024**	**0.0026**	**0.0017**	**0.0024**	**0.0017**	**0.0018**	**0.0027**	**0.0022**
	R	**0.0159**	**0.0140**	**0.0143**	**0.0131**	**0.0139**	**0.0143**	**0.0131**	**0.0132**	**0.0147**	**0.0140**	**0.0152**	**0.0142**	**0.0147**	**0.0150**	**0.0154**	**0.0143**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**16**	**8**	**4**	**6**	**10**	**3**	**2**	**6**	**7**	**7**	**9**	**7**	**2**	**13**	**3**	**7**
	R	**26**	**18**	**17**	**19**	**24**	**27**	**27**	**30**	**35**	**48**	**38**	**45**	**45**	**54**	**58**	**34**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**3685**	**2237**	**1204**	**1858**	**3087**	**1201**	**925**	**2092**	**2478**	**2858**	**3095**	**2920**	**1120**	**5128**	**1708**	**2373**
	R	**6019**	**4658**	**3354**	**4841**	**6264**	**7326**	**6891**	**8532**	**10845**	**14742**	**13468**	**15007**	**14987**	**18019**	**20418**	**10358**

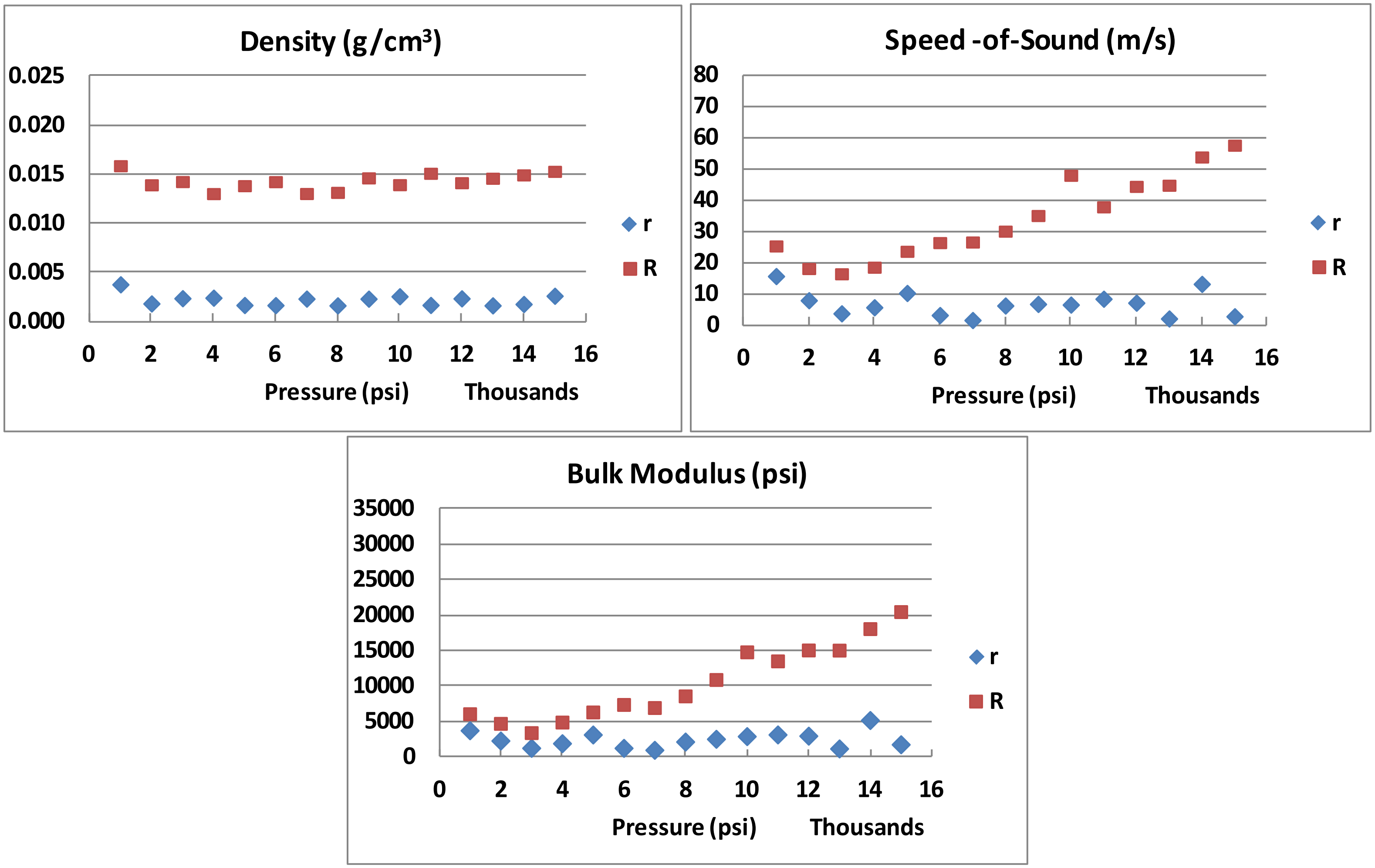


Figure A-4. Sample 6484 at 65 °C

Table A-5. Sample 6485 at 35 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0018**	**0.0018**	**0.0017**	**0.0019**	**0.0005**	**0.0009**	**0.0010**	**0.0008**	**0.0025**	**0.0008**	**0.0008**	**0.0006**	**0.0019**	**0.0008**	**0.0007**	**0.0012**
	R	**0.0131**	**0.0122**	**0.0123**	**0.0126**	**0.0119**	**0.0122**	**0.0121**	**0.0134**	**0.0120**	**0.0117**	**0.0129**	**0.0131**	**0.0135**	**0.0139**	**0.0163**	**0.0129**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**45**	**6**	**33**	**7**	**8**	**21**	**11**	**26**	**11**	**13**	**15**	**13**	**10**	**8**	**6**	**15**
	R	**60**	**38**	**39**	**35**	**38**	**53**	**57**	**51**	**45**	**60**	**48**	**74**	**64**	**74**	**60**	**53**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**15192**	**2166**	**11192**	**2451**	**3074**	**7601**	**4213**	**10216**	**4319**	**4912**	**5868**	**5309**	**4096**	**2998**	**2417**	**5735**
	R	**18259**	**12270**	**13596**	**10966**	**11592**	**20131**	**21283**	**20032**	**17325**	**22347**	**19734**	**31536**	**26199**	**32407**	**27913**	**20373**

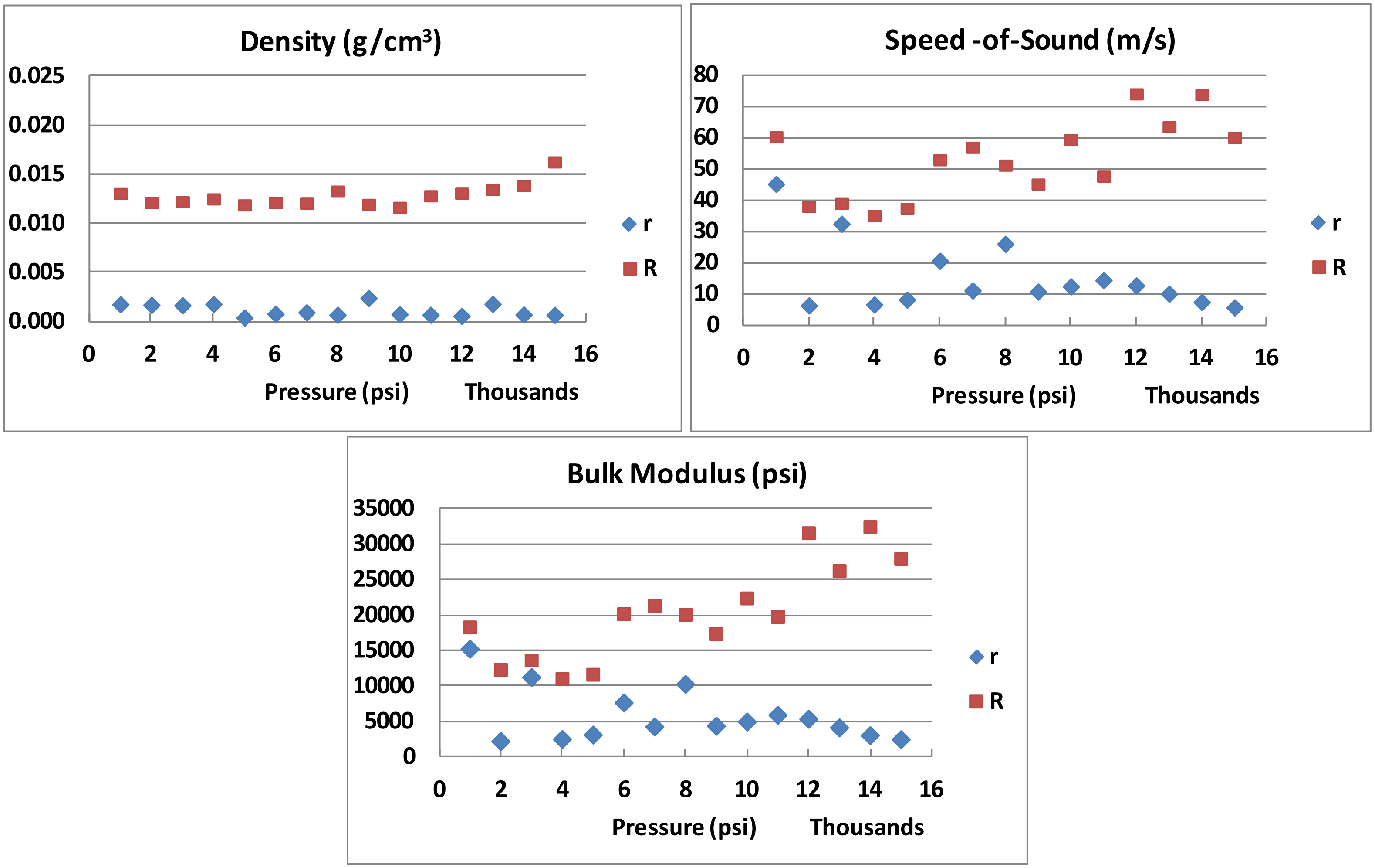


Figure A-5. Sample 6485 at 35 °C

Table A-6. Sample 6485 at 65 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0021**	**0.0013**	**0.0012**	**0.0012**	**0.0028**	**0.0007**	**0.0008**	**0.0012**	**0.0019**	**0.0007**	**0.0009**	**0.0008**	**0.0011**	**0.0020**	**0.0011**	**0.0013**
	R	**0.0151**	**0.0151**	**0.0151**	**0.0122**	**0.0155**	**0.0162**	**0.0155**	**0.0115**	**0.0145**	**0.0151**	**0.0183**	**0.0127**	**0.0152**	**0.0148**	**0.0125**	**0.0146**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**15**	**14**	**17**	**12**	**9**	**5**	**1**	**11**	**10**	**10**	**9**	**12**	**6**	**6**	**6**	**10**
	R	**24**	**22**	**30**	**24**	**29**	**36**	**34**	**44**	**54**	**48**	**45**	**33**	**63**	**56**	**47**	**39**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**4198**	**4321**	**5388**	**4011**	**3068**	**1769**	**576**	**4039**	**3781**	**3669**	**3429**	**5014**	**2691**	**2436**	**2375**	**3384**
	R	**7047**	**6211**	**7876**	**7316**	**9423**	**10575**	**11219**	**15974**	**16873**	**15946**	**12210**	**11976**	**23478**	**20674**	**20869**	**13178**

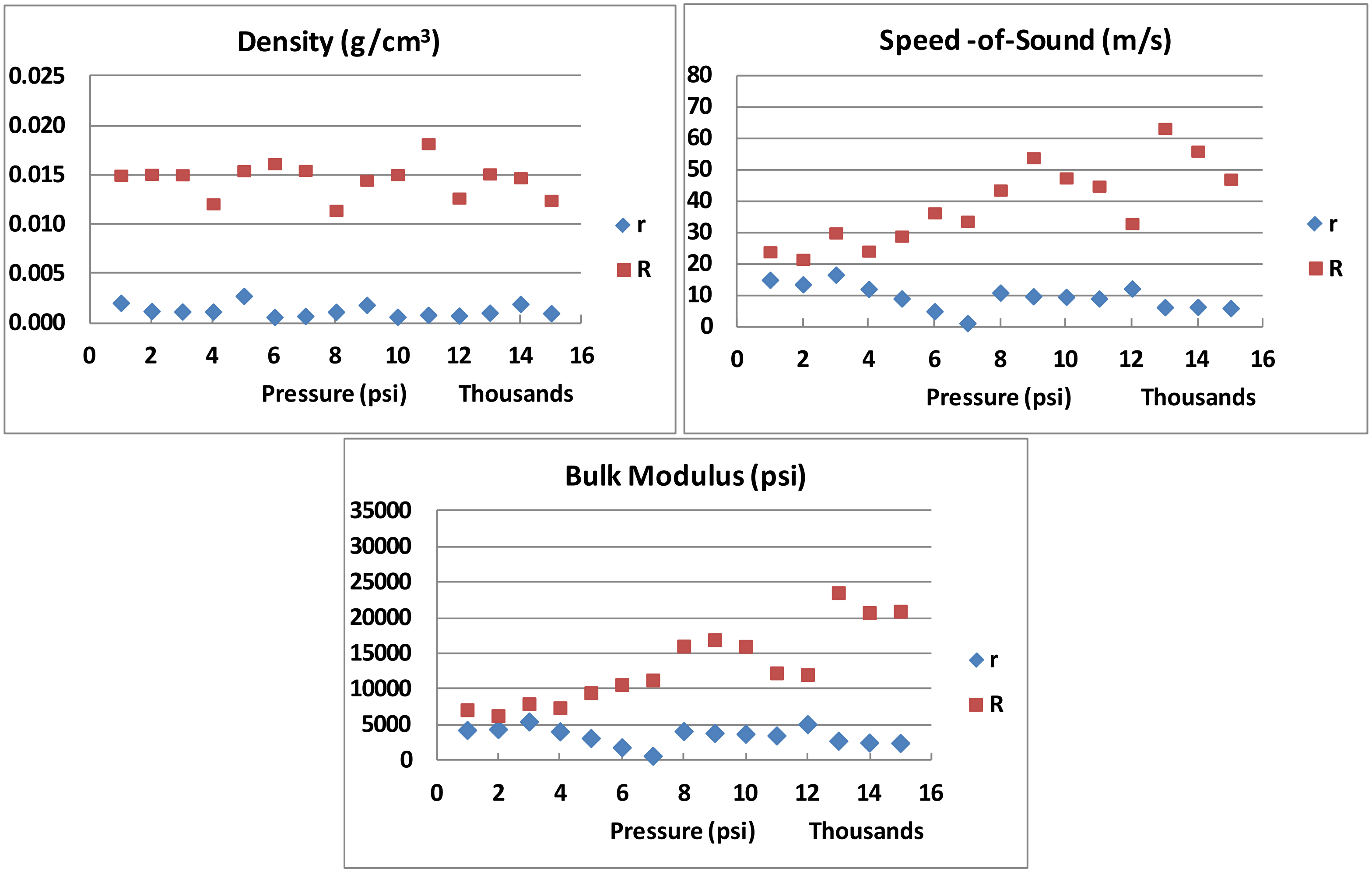


Figure A-6. Sample 6485 at 65 °C

Table A-7. Sample 6486 at 35 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0066**	**0.0030**	**0.0036**	**0.0034**	**0.0025**	**0.0028**	**0.0019**	**0.0022**	**0.0021**	**0.0020**	**0.0003**	**0.0006**	**0.0005**	**0.0019**	**0.0007**	**0.0023**
	R	**0.0220**	**0.0189**	**0.0187**	**0.0188**	**0.0190**	**0.0186**	**0.0179**	**0.0184**	**0.0175**	**0.0179**	**0.0173**	**0.0165**	**0.0169**	**0.0175**	**0.0176**	**0.0182**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**7**	**4**	**15**	**14**	**8**	**13**	**9**	**7**	**16**	**15**	**3**	**3**	**9**	**14**	**10**	**10**
	R	**22**	**24**	**23**	**25**	**26**	**28**	**28**	**29**	**32**	**43**	**37**	**34**	**42**	**47**	**57**	**33**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**3282**	**978**	**4945**	**4837**	**2511**	**4904**	**3402**	**2491**	**5652**	**5384**	**1143**	**1023**	**3432**	**5370**	**4178**	**3569**
	R	**6877**	**6842**	**6752**	**6715**	**7911**	**10054**	**6887**	**6654**	**12338**	**14348**	**12502**	**10308**	**15640**	**17547**	**22503**	**10925**

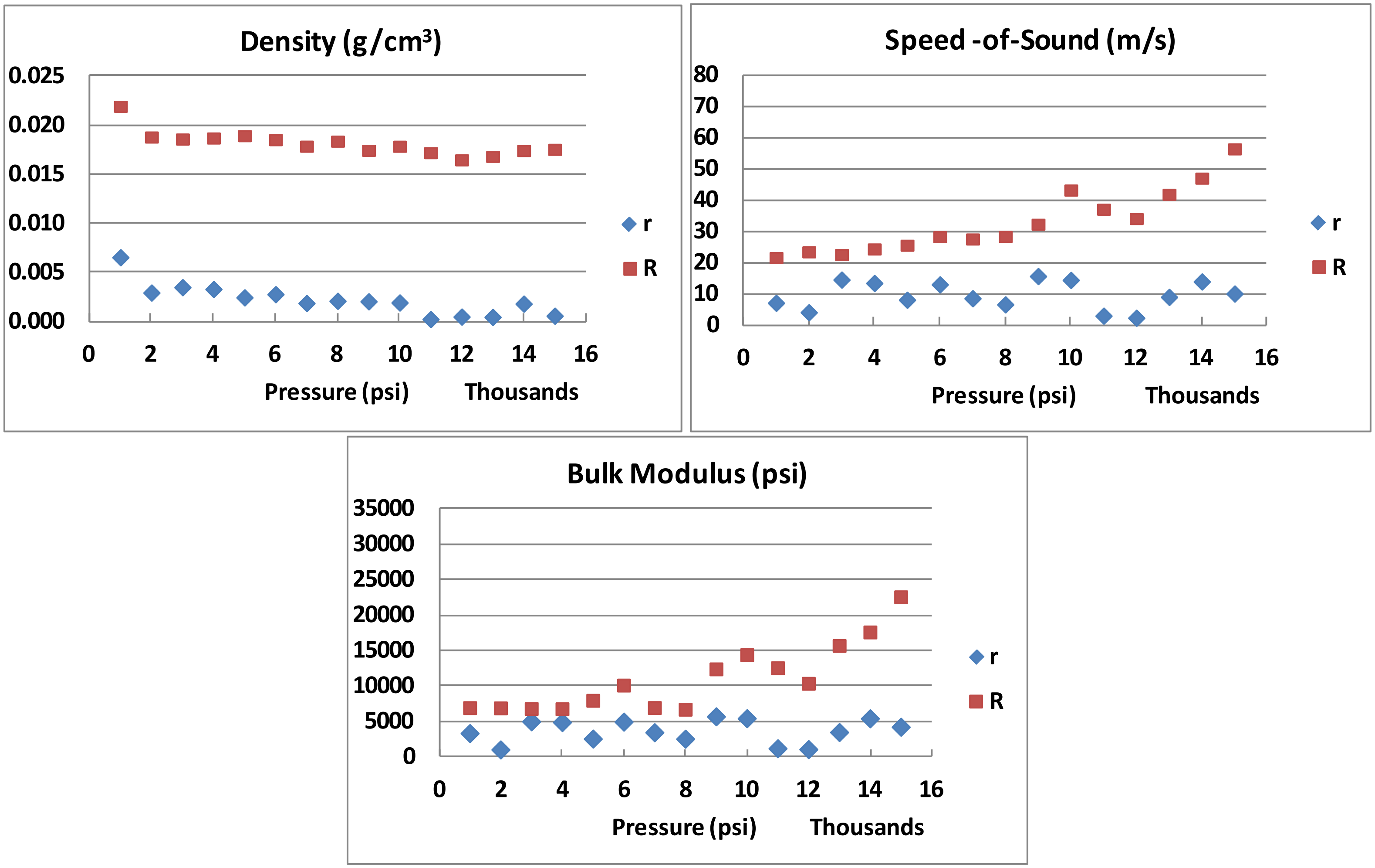


Figure A-7. Sample 6486 at 35 °C

Table A-8. Sample 6486 at 65 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0018**	**0.0012**	**0.0019**	**0.0008**	**0.0006**	**0.0021**	**0.0008**	**0.0005**	**0.0005**	**0.0017**	**0.0018**	**0.0017**	**0.0010**	**0.0022**	**0.0018**	**0.0014**
	R	**0.0198**	**0.0192**	**0.0206**	**0.0212**	**0.0192**	**0.0203**	**0.0193**	**0.0187**	**0.0201**	**0.0194**	**0.0188**	**0.0178**	**0.0191**	**0.0192**	**0.0210**	**0.0196**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**10**	**9**	**10**	**7**	**14**	**8**	**16**	**14**	**13**	**16**	**10**	**3**	**10**	**13**	**12**	**11**
	R	**25**	**19**	**17**	**21**	**13**	**22**	**23**	**24**	**22**	**26**	**25**	**39**	**38**	**45**	**47**	**27**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**2190**	**2348**	**2611**	**2029**	**3950**	**2764**	**4854**	**4217**	**4227**	**5265**	**3634**	**1190**	**3531**	**4416**	**4283**	**3434**
	R	**8075**	**7528**	**5589**	**7277**	**6586**	**5930**	**7890**	**10236**	**5157**	**10110**	**7530**	**13250**	**13763**	**13806**	**16064**	**9253**

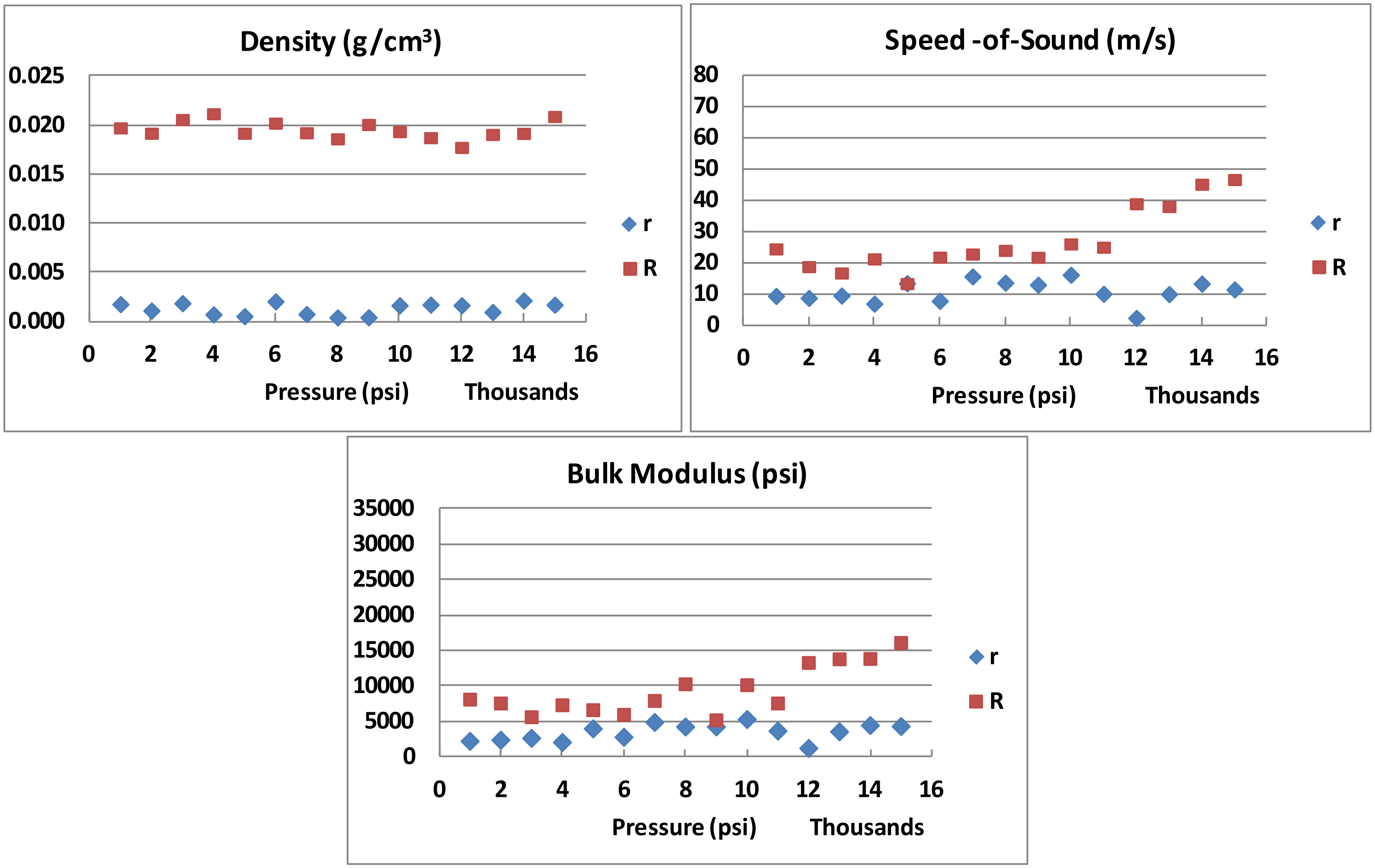


Figure A-8. Sample 6486 at 65 °C

Table A-9. Sample 6487 at 35 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0014**	**0.0007**	**0.0003**	**0.0005**	**0.0018**	**0.0018**	**0.0007**	**0.0007**	**0.0006**	**0.0007**	**0.0018**	**0.0010**	**0.0009**	**0.0006**	**0.0006**	**0.0010**
	R	**0.0134**	**0.0114**	**0.0113**	**0.0110**	**0.0114**	**0.0122**	**0.0112**	**0.0123**	**0.0112**	**0.0112**	**0.0117**	**0.0131**	**0.0129**	**0.0146**	**0.0172**	**0.0124**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**14**	**14**	**8**	**7**	**10**	**8**	**23**	**14**	**11**	**5**	**12**	**5**	**16**	**11**	**6**	**11**
	R	**31**	**27**	**28**	**42**	**31**	**42**	**56**	**38**	**55**	**49**	**59**	**54**	**60**	**64**	**57**	**46**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**4086**	**4378**	**2362**	**2393**	**3215**	**2853**	**7935**	**5238**	**3981**	**1959**	**4649**	**2014**	**6651**	**4616**	**2501**	**3922**
	R	**8312**	**8191**	**7990**	**12867**	**9831**	**14340**	**18122**	**13561**	**19310**	**17968**	**22732**	**21492**	**24590**	**26744**	**28426**	**16965**

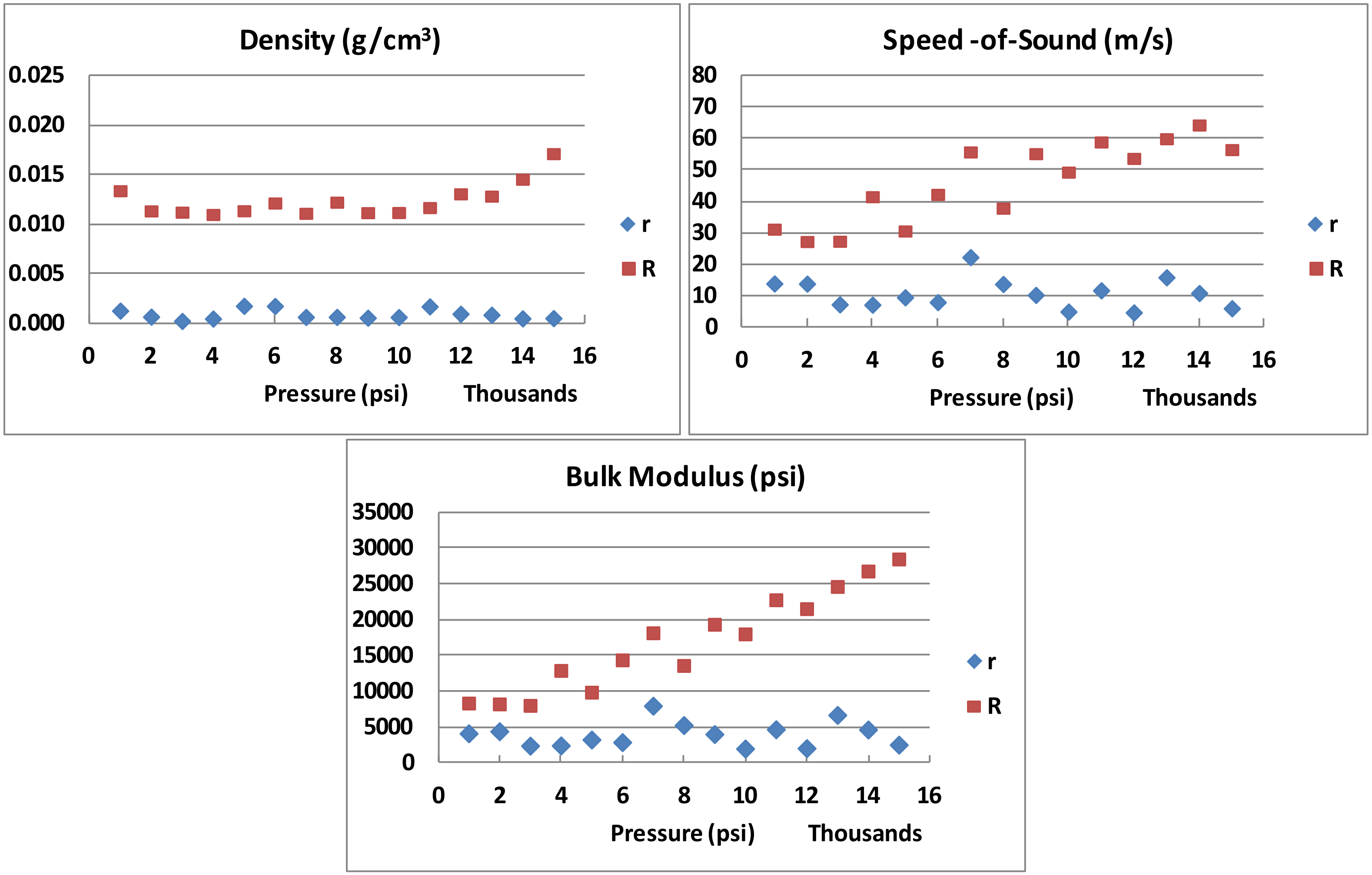


Figure A-9. Sample 6487 at 35 °C

Table A-10. Sample 6487 at 65 °C

Density	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
g/cm^3	**r**	**0.0026**	**0.0006**	**0.0017**	**0.0008**	**0.0018**	**0.0007**	**0.0005**	**0.0004**	**0.0004**	**0.0005**	**0.0004**	**0.0003**	**0.0017**	**0.0017**	**0.0006**	**0.0010**
	R	**0.0160**	**0.0156**	**0.0160**	**0.0148**	**0.0148**	**0.0167**	**0.0166**	**0.0152**	**0.0149**	**0.0167**	**0.0165**	**0.0168**	**0.0154**	**0.0161**	**0.0167**	**0.0159**
Spd of Snd	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
m/s	**r**	**8**	**6**	**5**	**14**	**8**	**21**	**6**	**5**	**12**	**10**	**7**	**8**	**10**	**15**	**9**	**10**
	R	**18**	**19**	**13**	**28**	**26**	**35**	**35**	**32**	**29**	**42**	**44**	**45**	**47**	**56**	**60**	**35**
Bulk Mod	psi	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000	11000	12000	13000	14000	15000	**Avg**
psi	**r**	**2066**	**1606**	**1569**	**4422**	**2719**	**6316**	**1886**	**1657**	**4063**	**3402**	**2534**	**2904**	**3681**	**5936**	**3484**	**3216**
	R	**5652**	**5318**	**4466**	**8279**	**6987**	**9546**	**10278**	**9249**	**10078**	**13491**	**15882**	**14823**	**16832**	**20223**	**22481**	**11572**

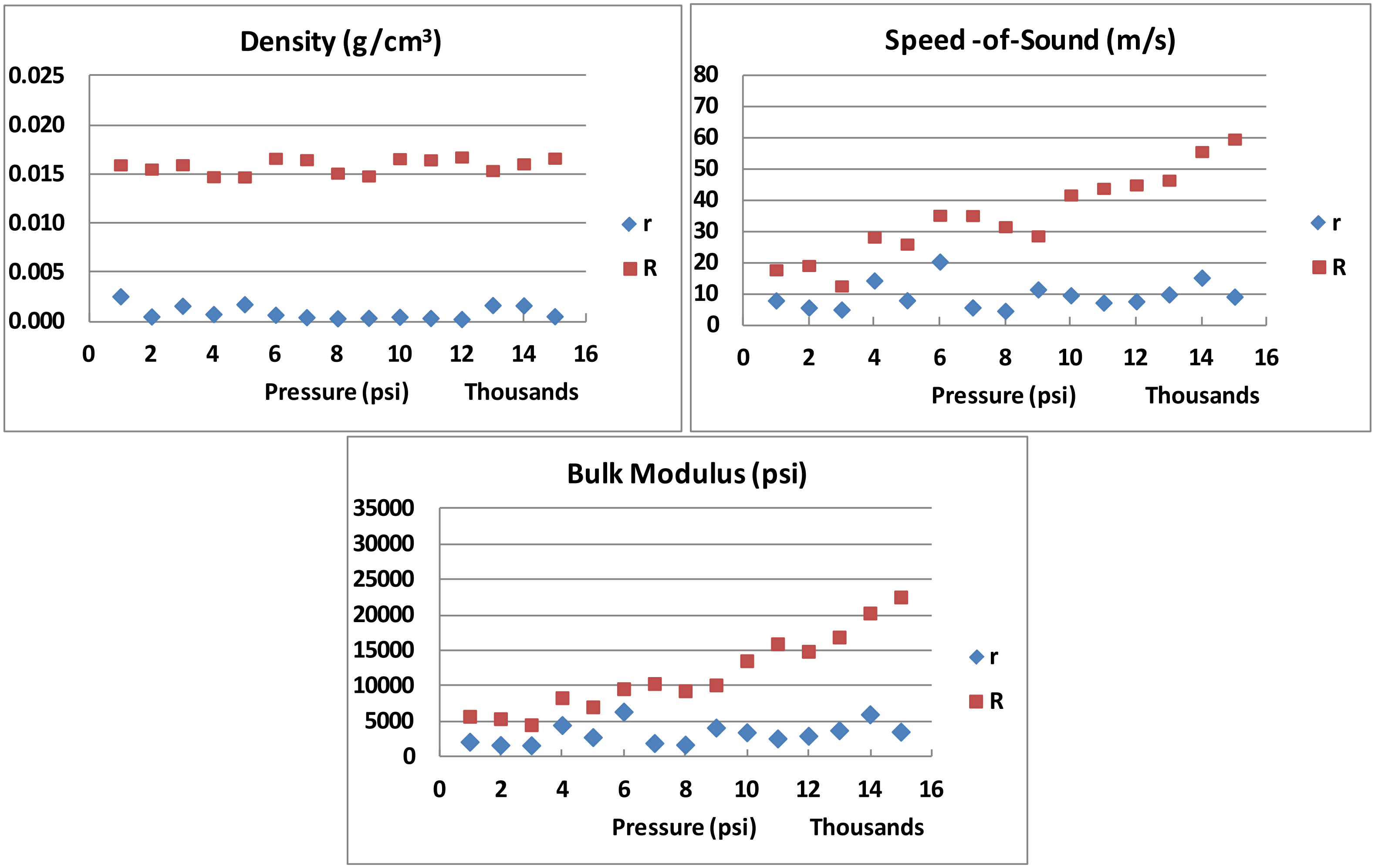


Figure A-10. Sample 6487 at 65 °C

APPENDIX B.

Temperature Comparison – Sample 6065

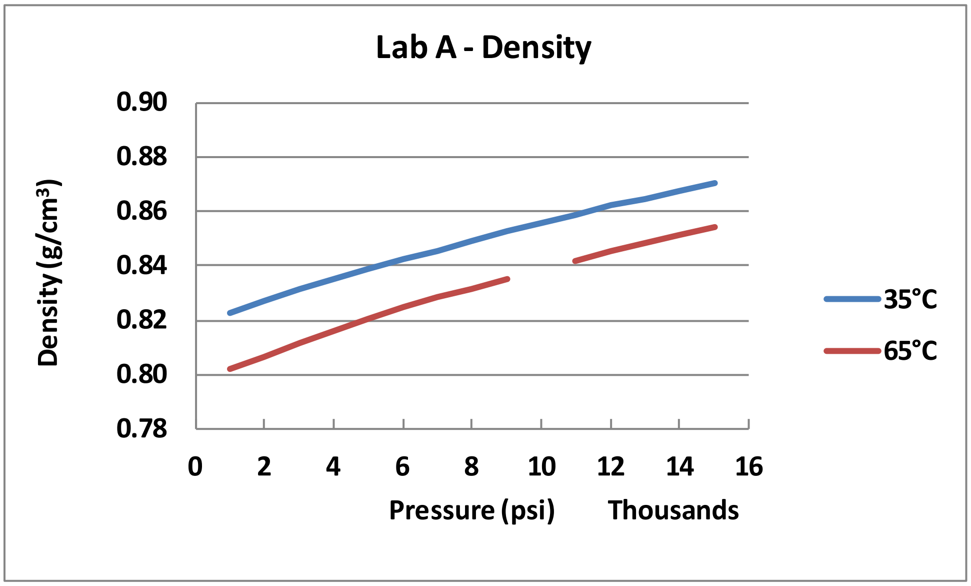


Figure B-1. Sample 6065, Lab A, Density

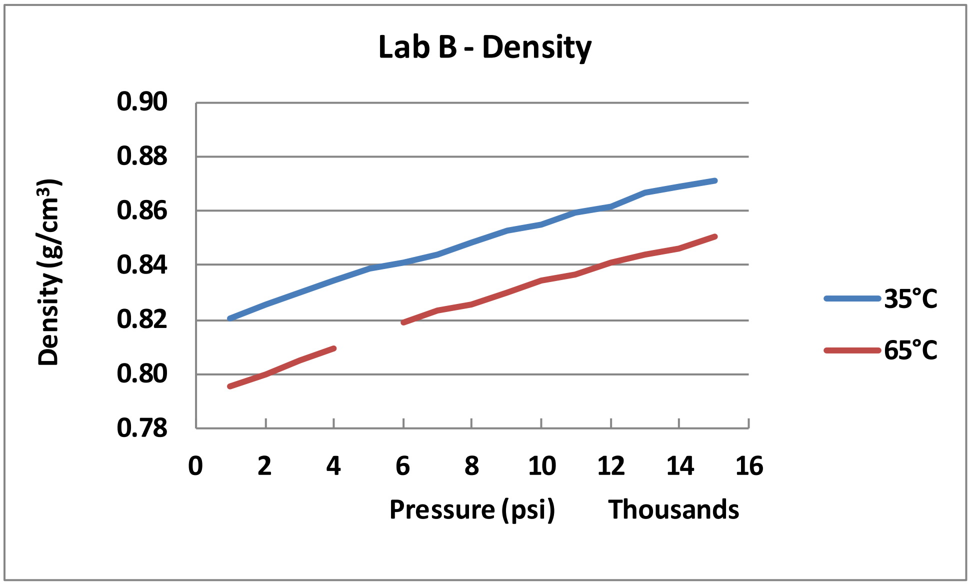


Figure B-2. Sample 6065, Lab B, Density

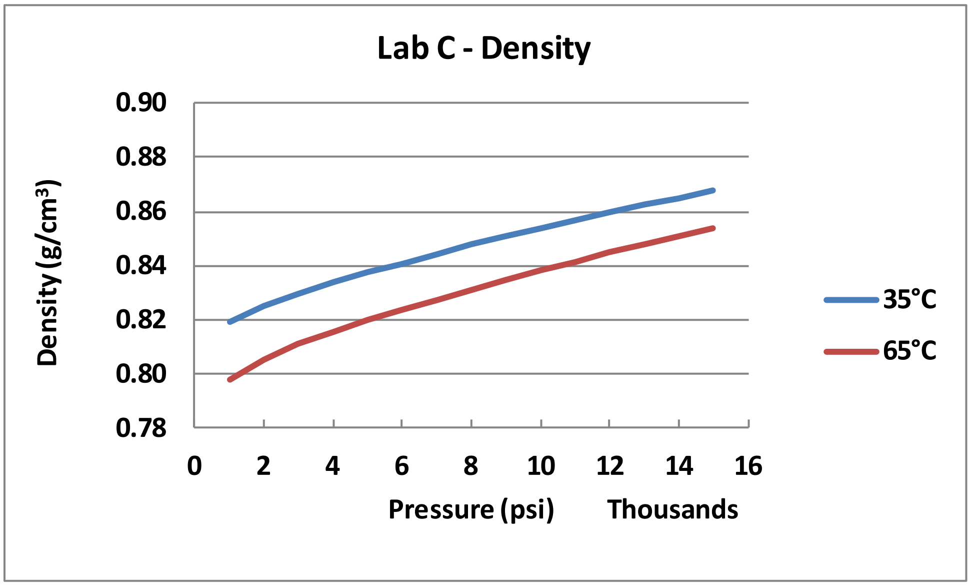


Figure B-3. Sample 6065, Lab C, Density

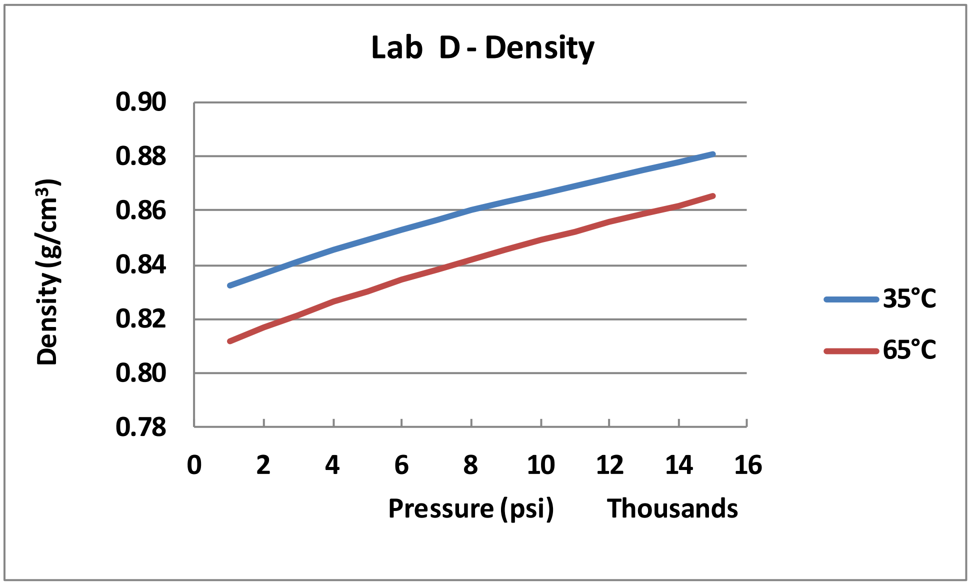


Figure B-4. Sample 6065, Lab D, Density

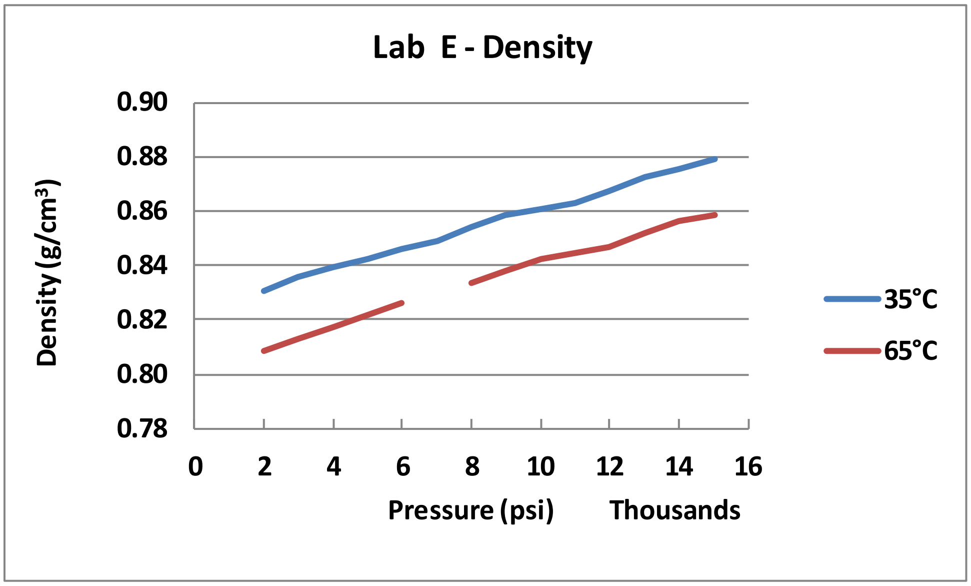


Figure B-5. Sample 6065, Lab E, Density

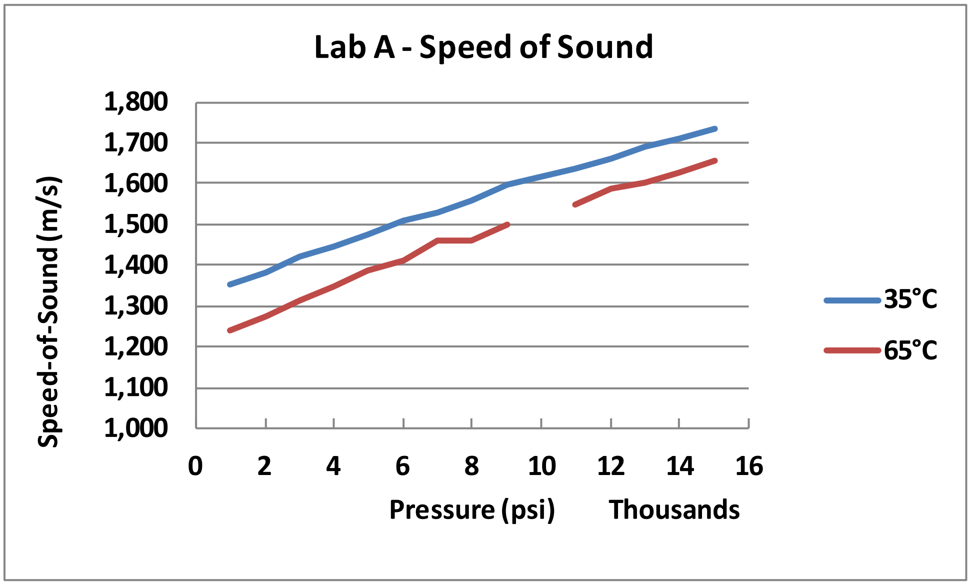


Figure B-6. Sample 6065, Lab A, Speed-of-Sound

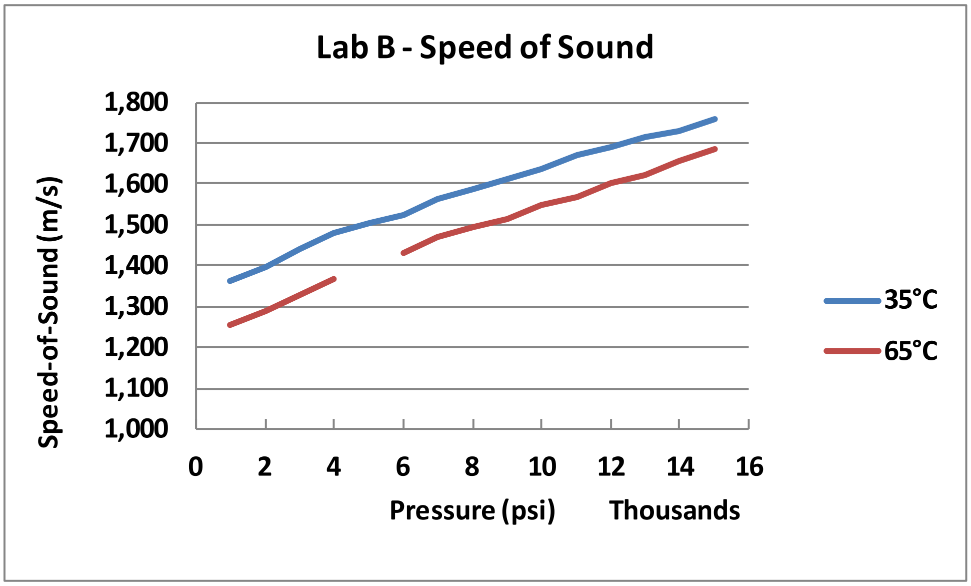


Figure B-7. Sample 6065, Lab B, Speed-of-Sound

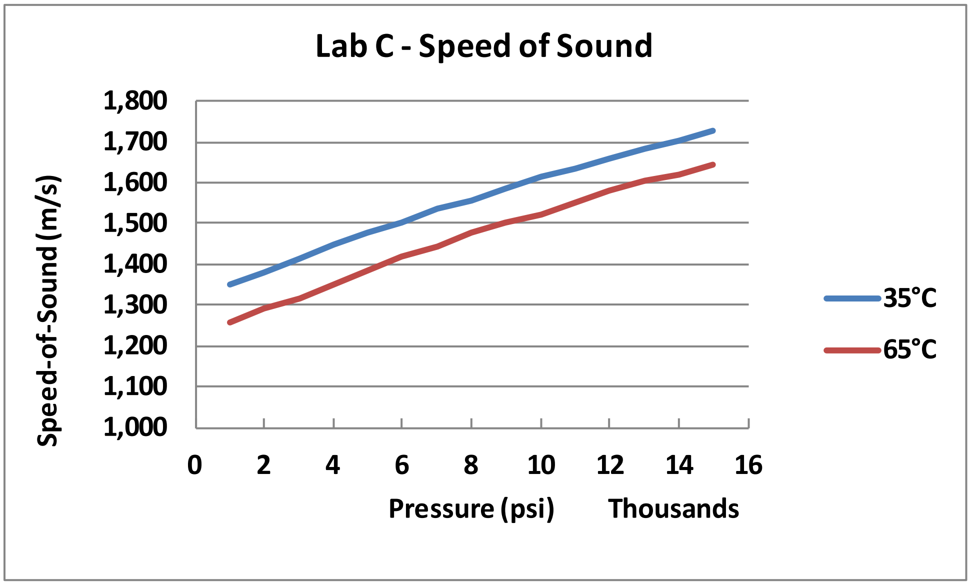


Figure B-8. Sample 6065, Lab C, Speed-of-Sound

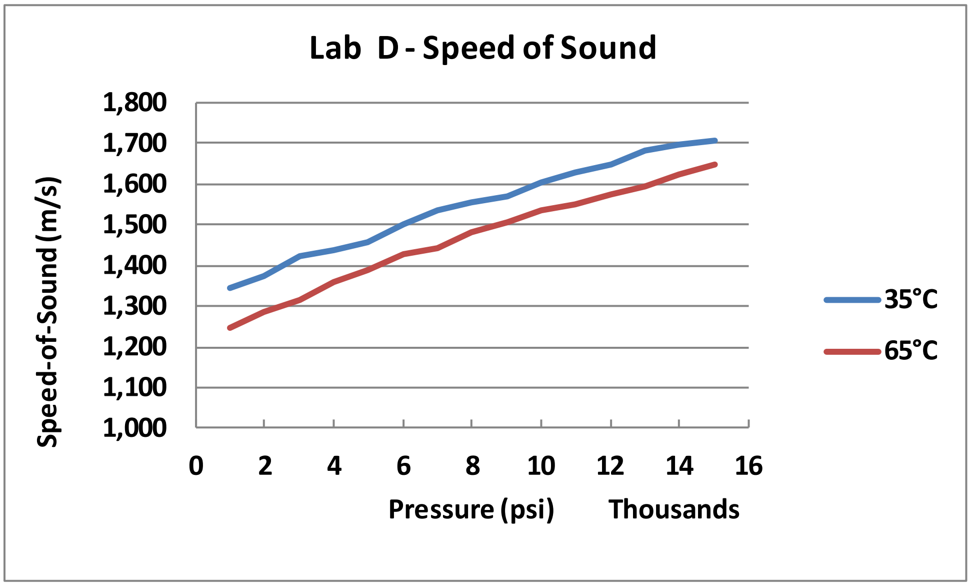


Figure B-9. Sample 6065, Lab D, Speed-of-Sound

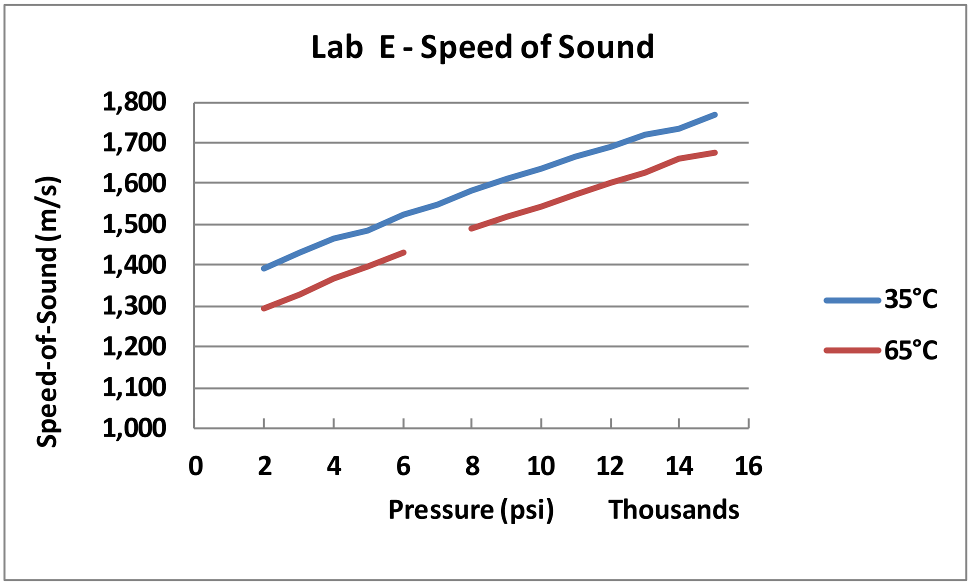


Figure B-10. Sample 6065, Lab E, Speed-of-Sound

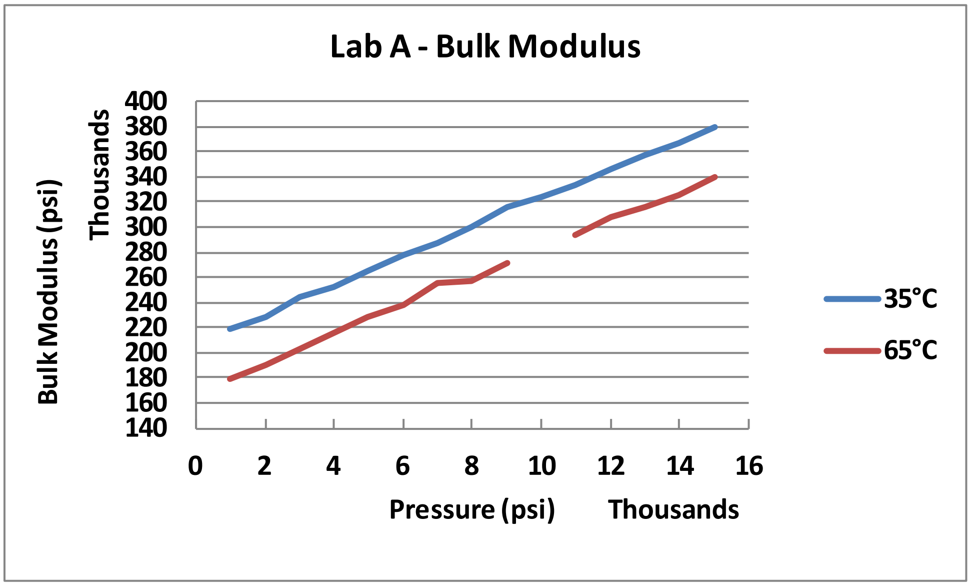


Figure B-11. Sample 6065, Lab A, Bulk Modulus

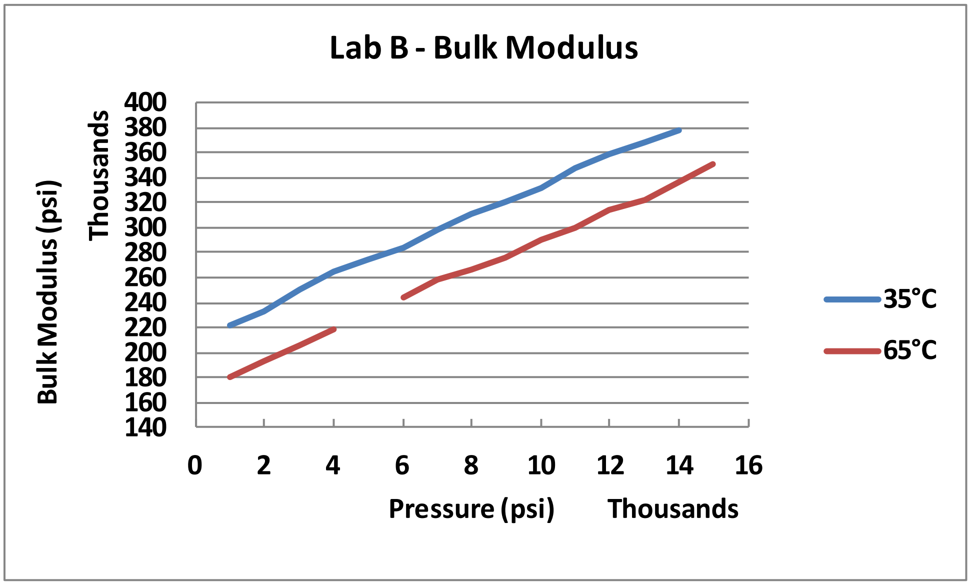


Figure B-12. Sample 6065, Lab B, Bulk Modulus

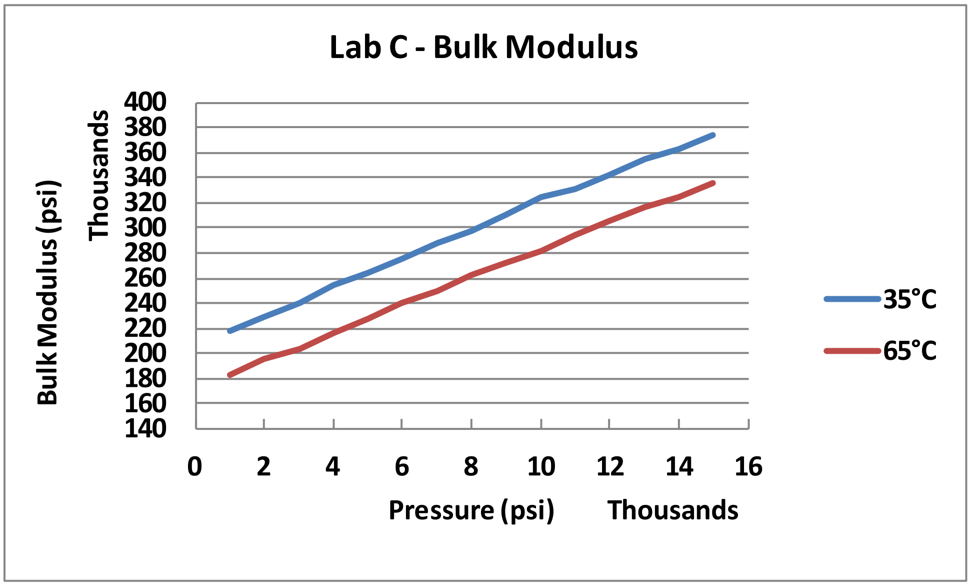


Figure B-13. Sample 6065, Lab C, Bulk Modulus

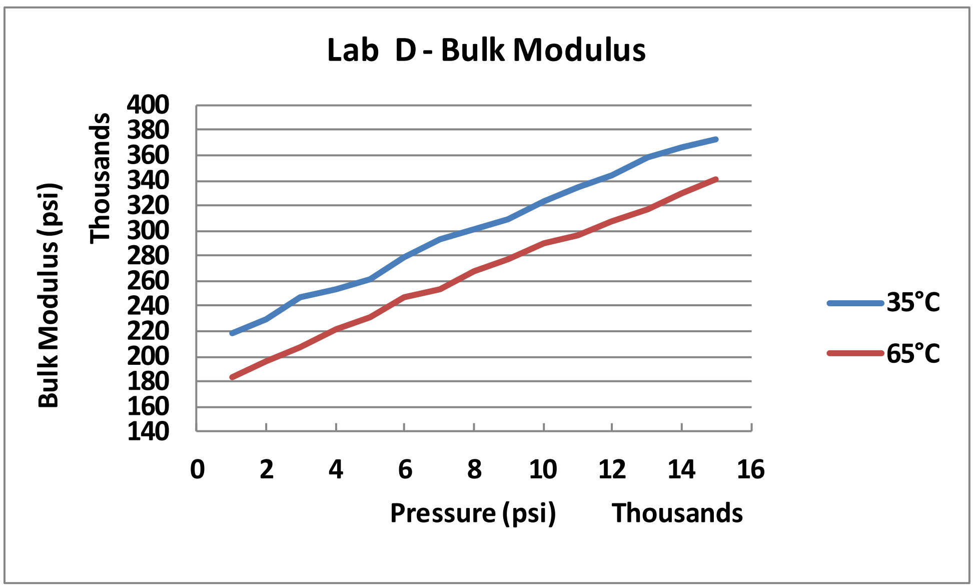


Figure B-14. Sample 6065, Lab D, Bulk Modulus

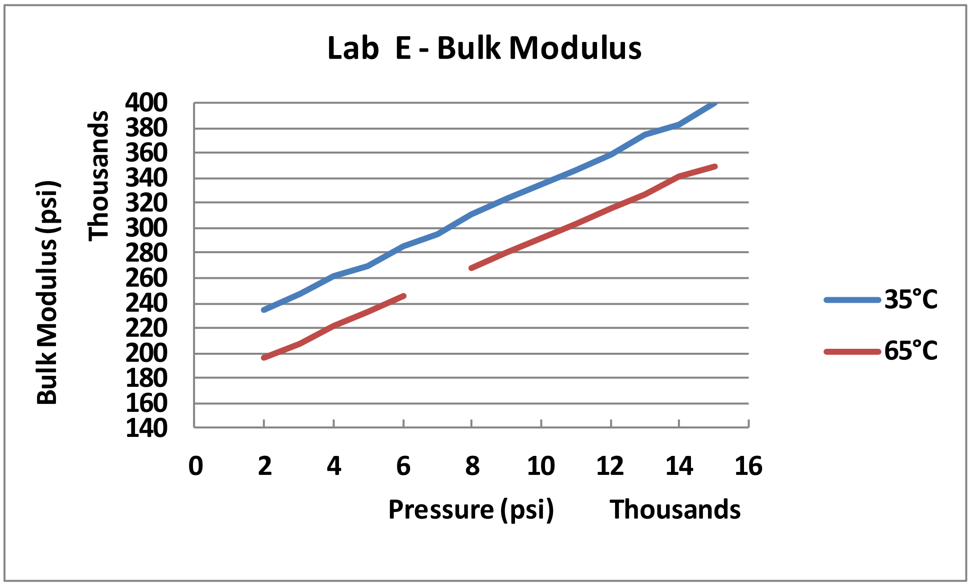


Figure B-15. Sample 6065, Lab E, Bulk Modulus

APPENDIX C.

Temperature Comparison – Sample 6484

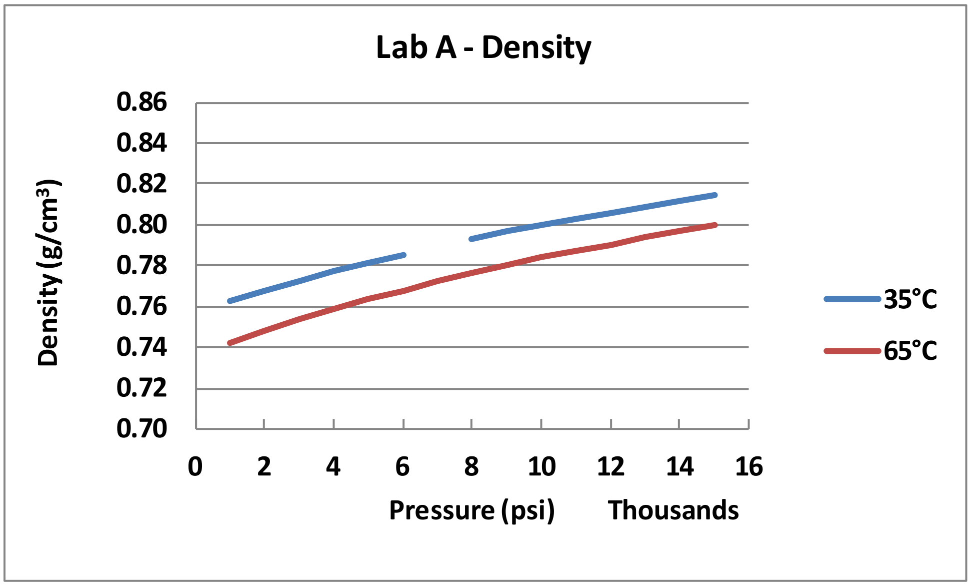


Figure C-1. Sample 6484, Lab A, Density

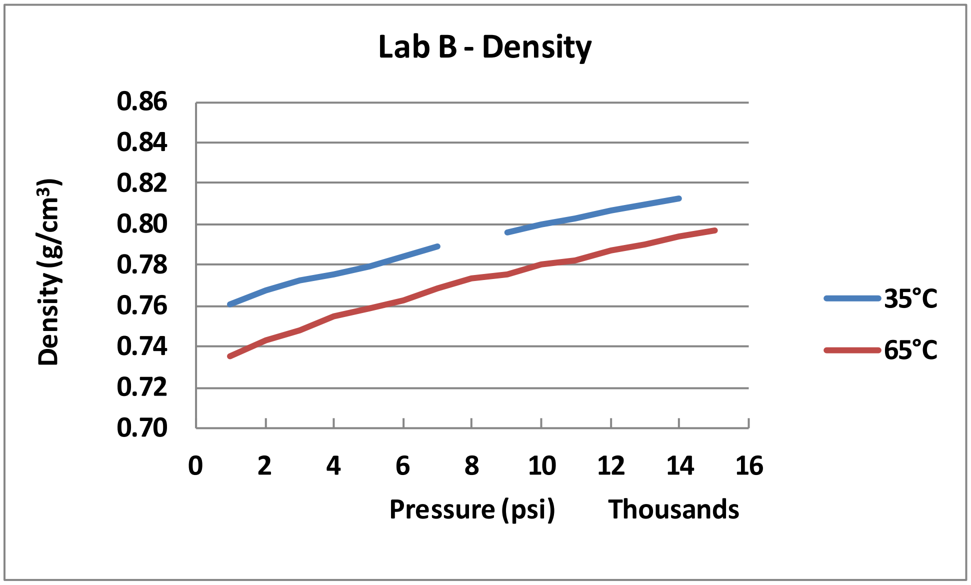


Figure C-2. Sample 6484, Lab B, Density

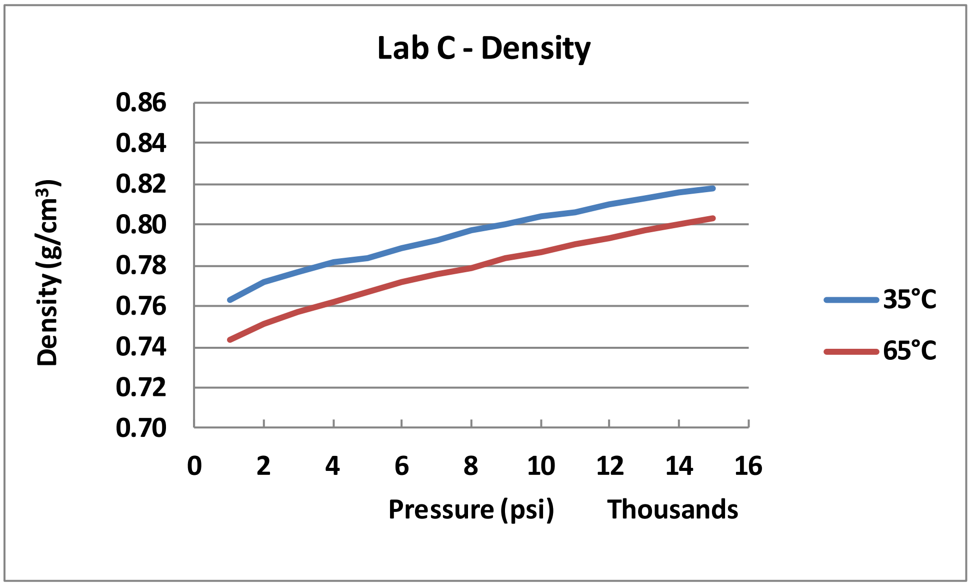


Figure C-3. Sample 6484, Lab C, Density

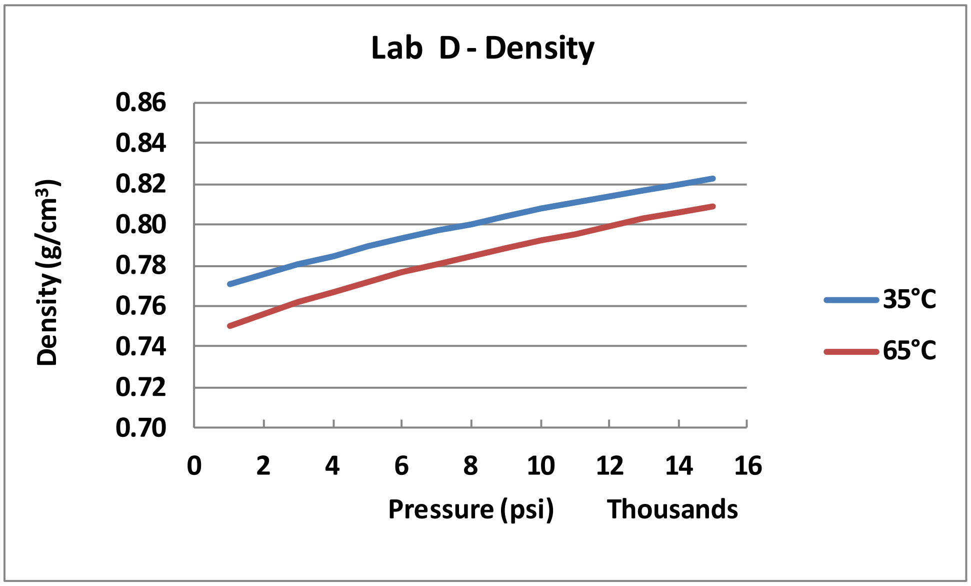


Figure C-4. Sample 6484, Lab D, Density

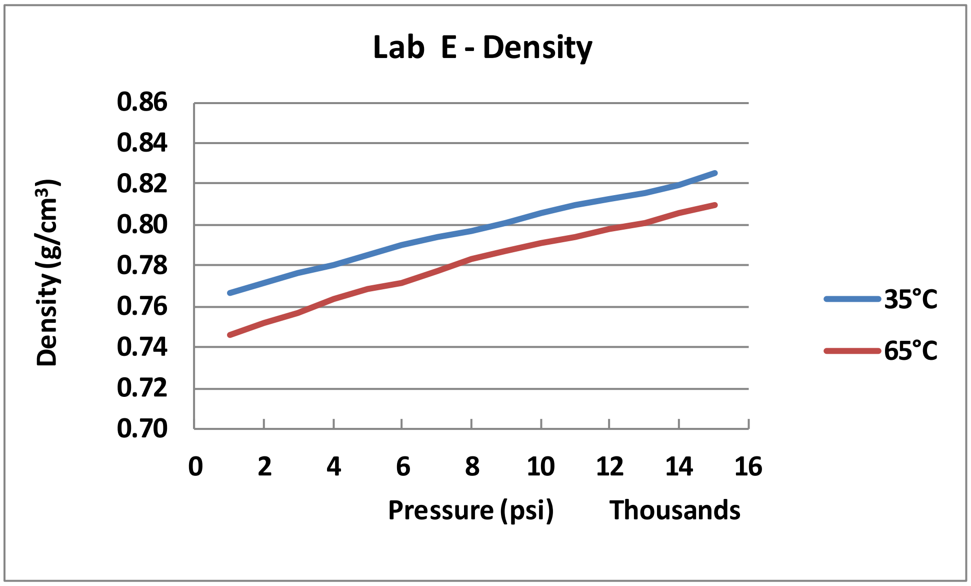


Figure C-5. Sample 6484, Lab E, Density

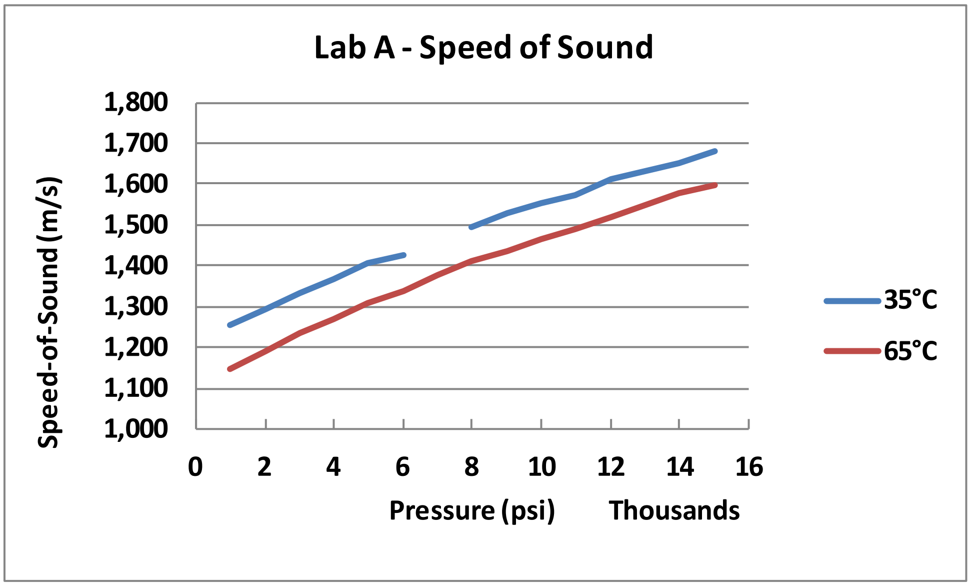


Figure C-6. Sample 6484, Lab A, Speed-of-Sound

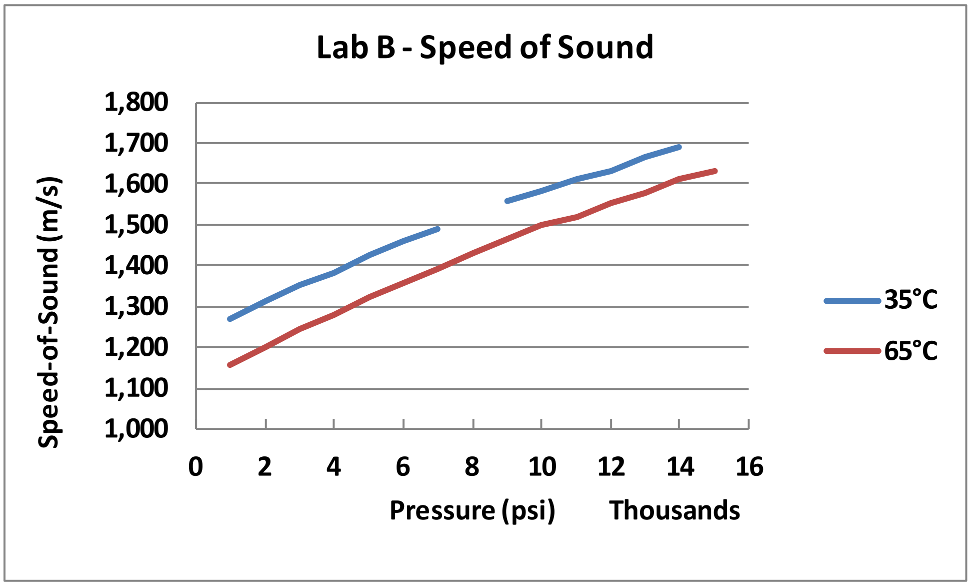


Figure C-7. Sample 6484, Lab B, Speed-of-Sound

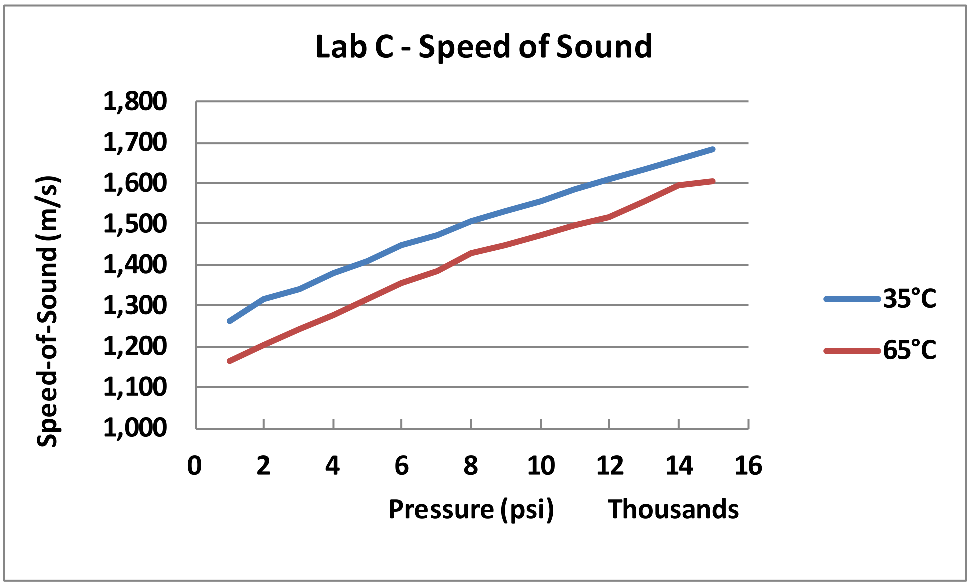


Figure C-8. Sample 6484, Lab C, Speed-of-Sound

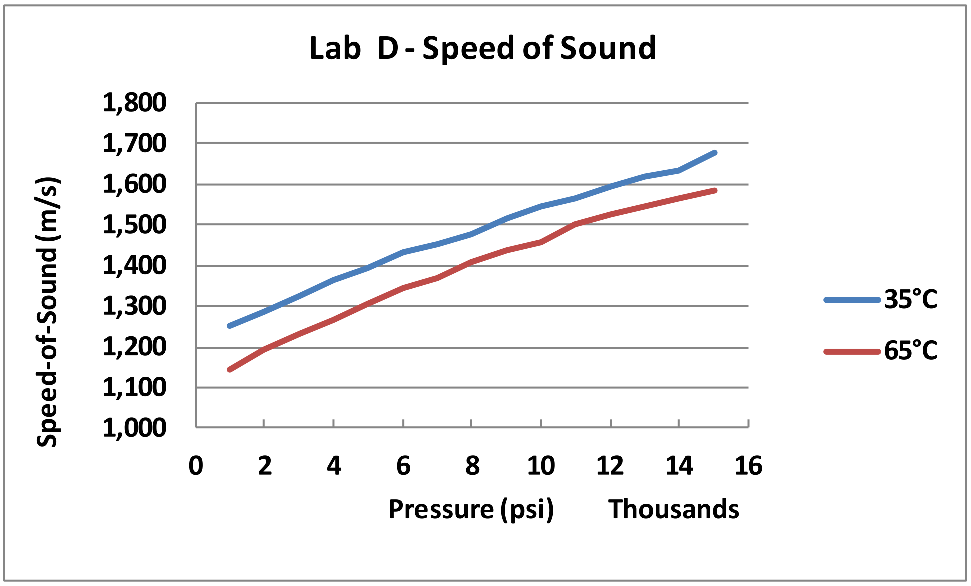


Figure C-9. Sample 6484, Lab D, Speed-of-Sound

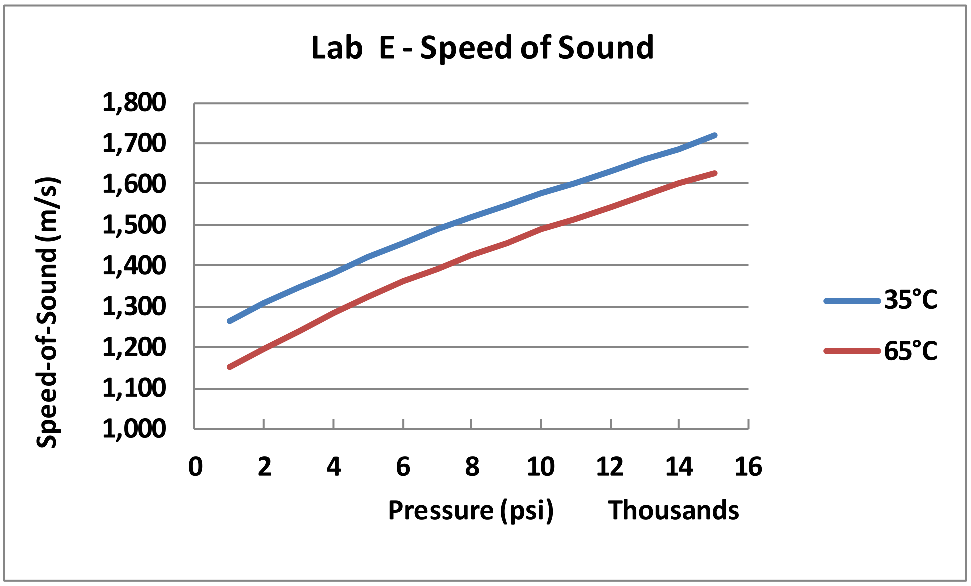


Figure C-10. Sample 6484, Lab E, Speed-of-Sound

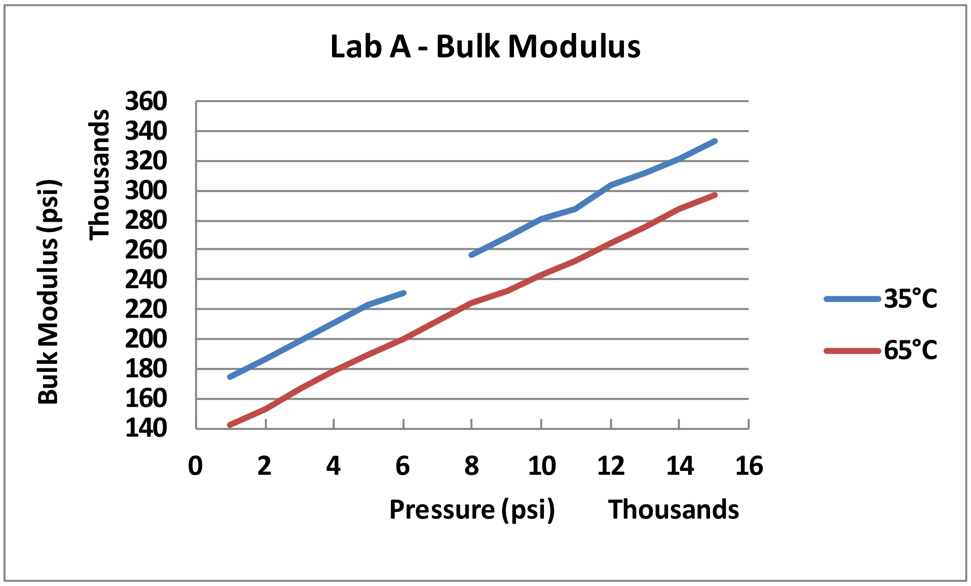


Figure C-11. Sample 6484, Lab A, Bulk Modulus

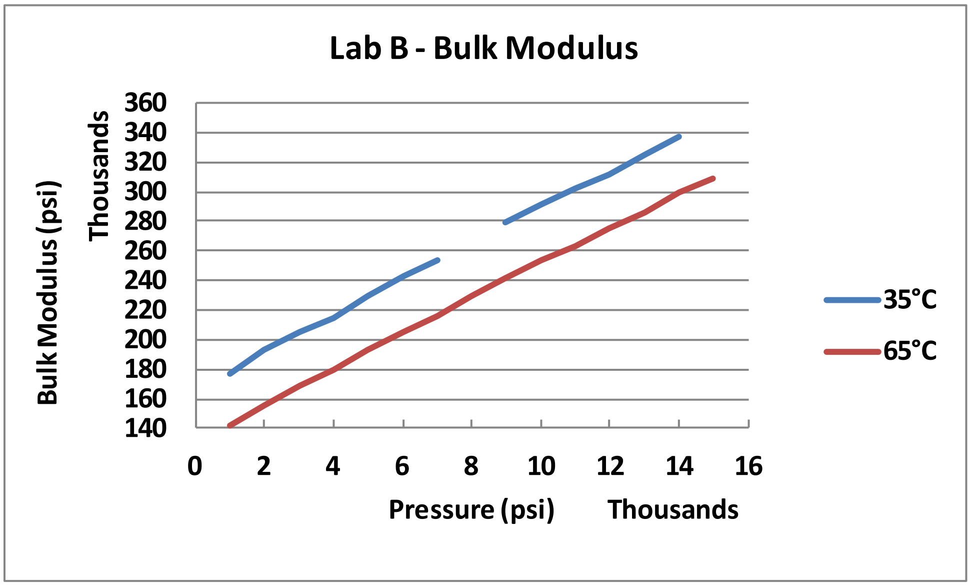


Figure C-12. Sample 6484, Lab B, Bulk Modulus

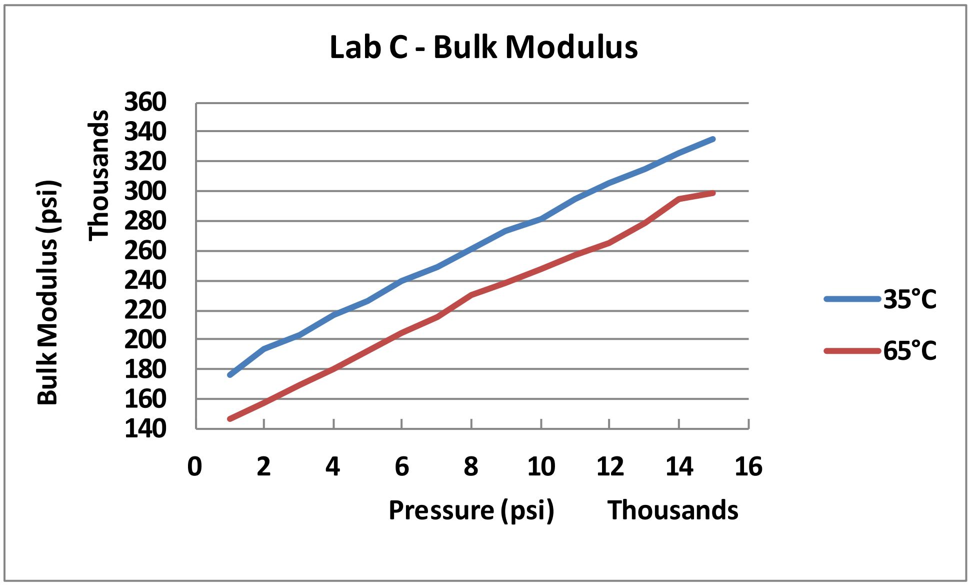


Figure C-13. Sample 6484, Lab C, Bulk Modulus

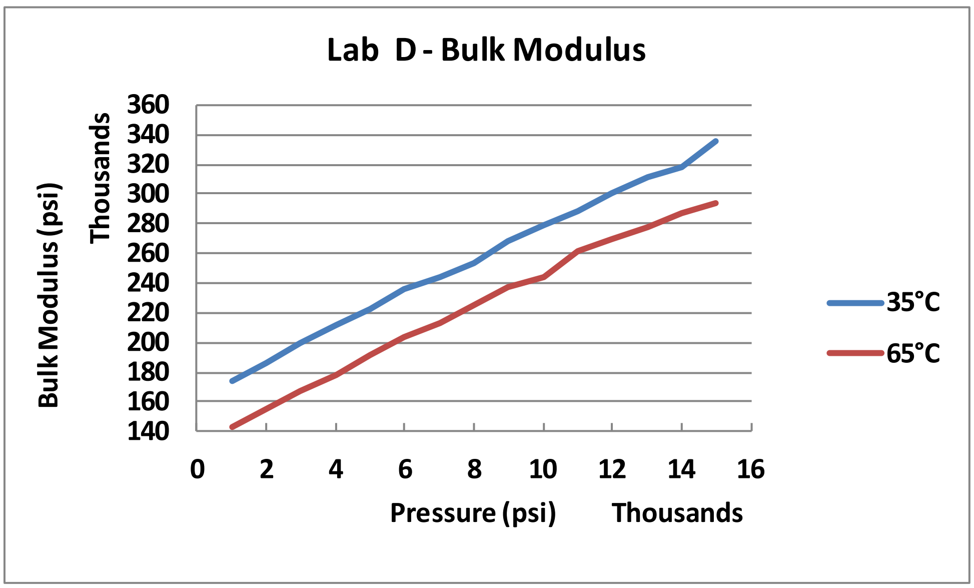


Figure C-14. Sample 6484, Lab D, Bulk Modulus

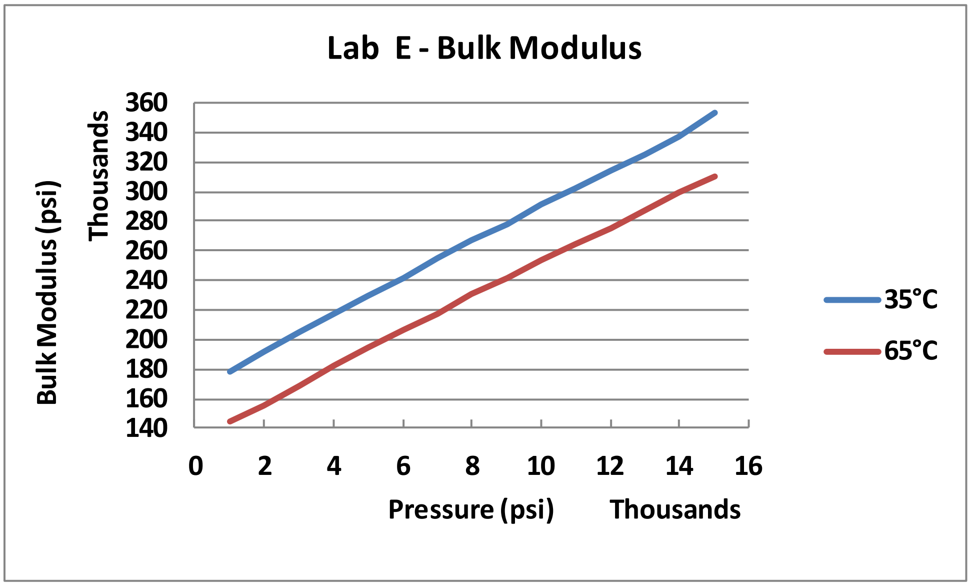


Figure C-15. Sample 6484, Lab E, Bulk Modulus

APPENDIX D.

Temperature Comparison – Sample 6485

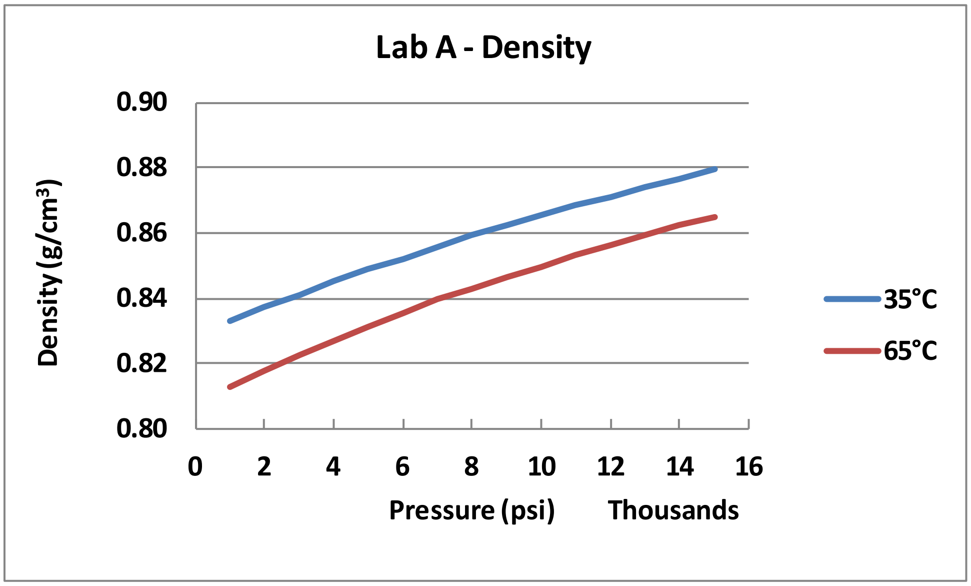


Figure D-1. Sample 6485, Lab A, Density

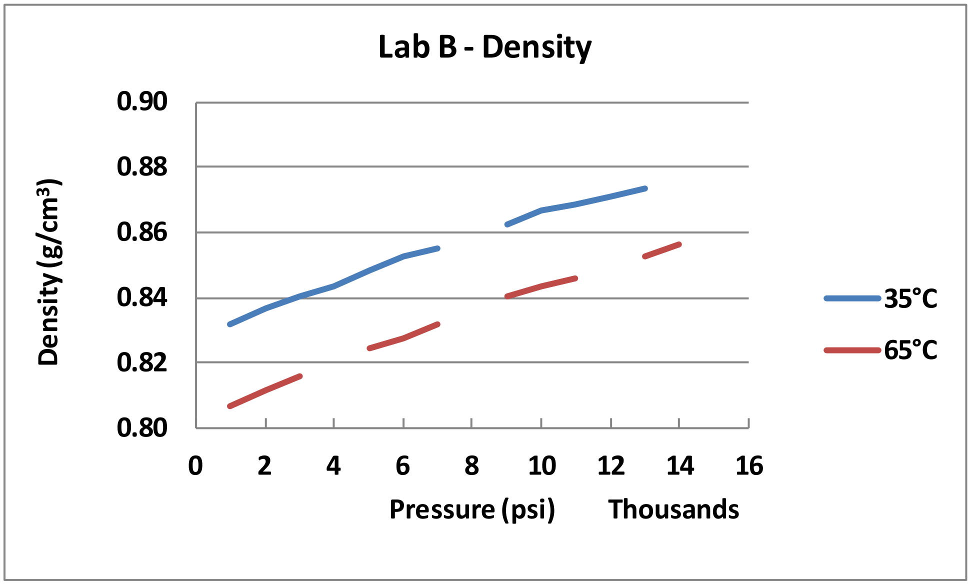


Figure D-2. Sample 6485, Lab B, Density

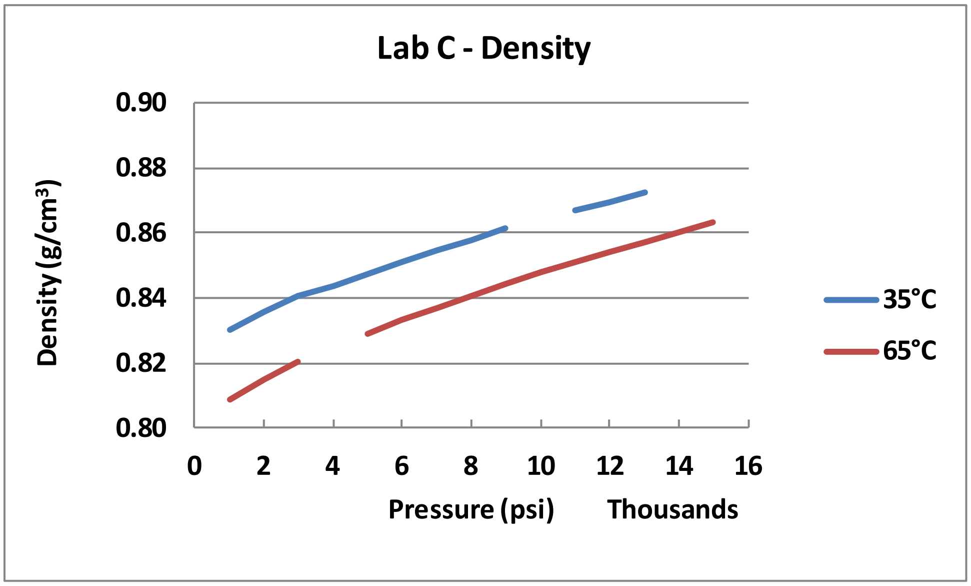


Figure D-3. Sample 6485, Lab C, Density

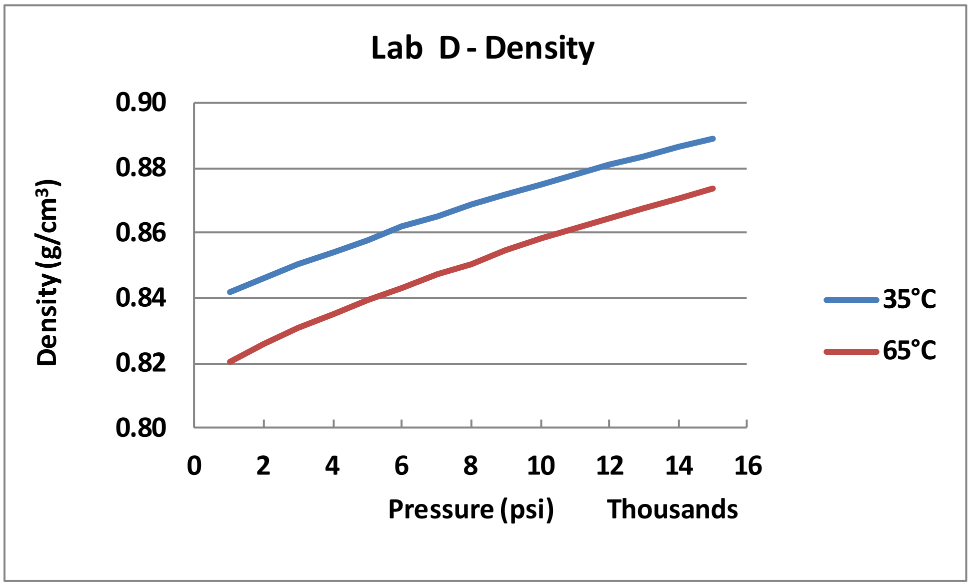


Figure D-4. Sample 6485, Lab D, Density

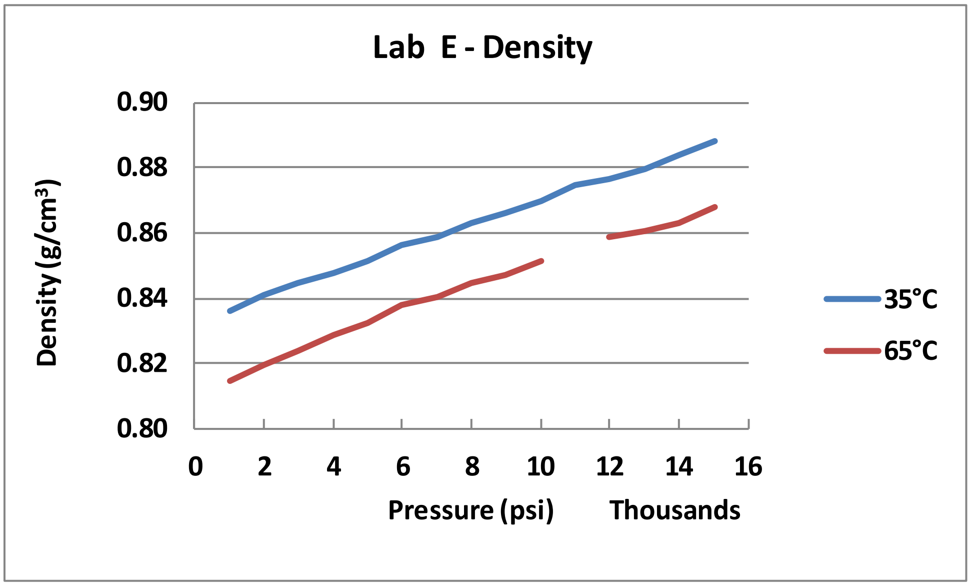


Figure D-5. Sample 6485, Lab E, Density

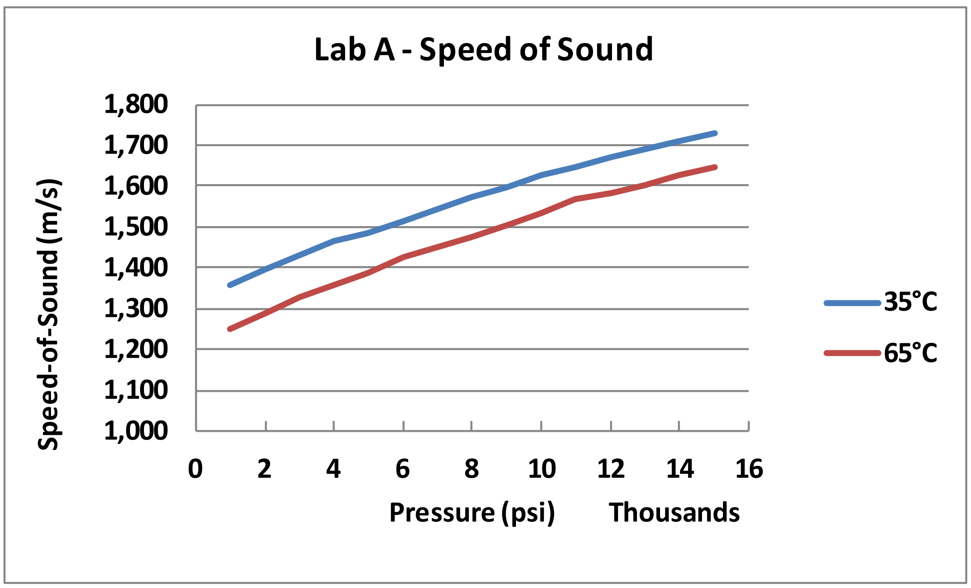


Figure D-6. Sample 6485, Lab A, Speed-of-Sound

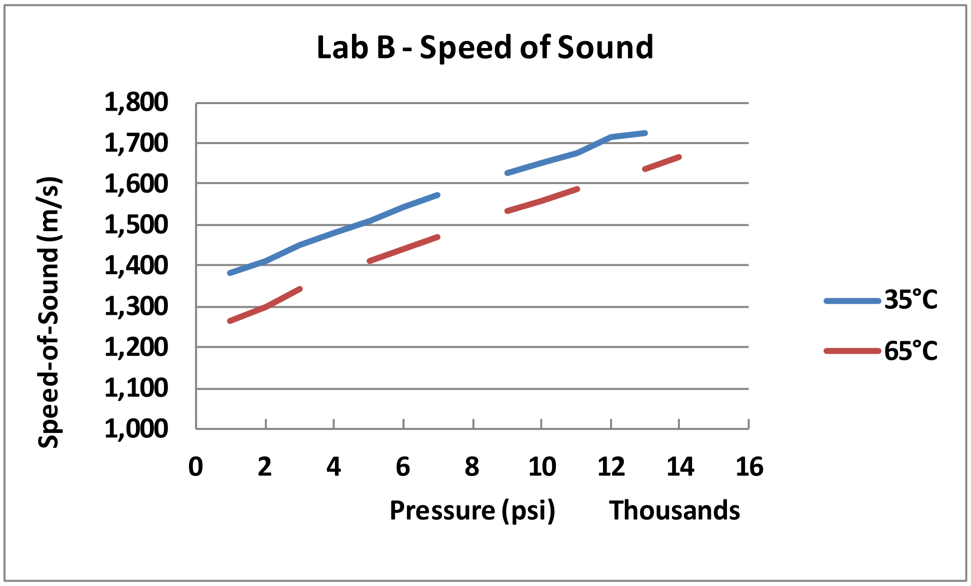


Figure D-7. Sample 6485, Lab B, Speed-of-Sound

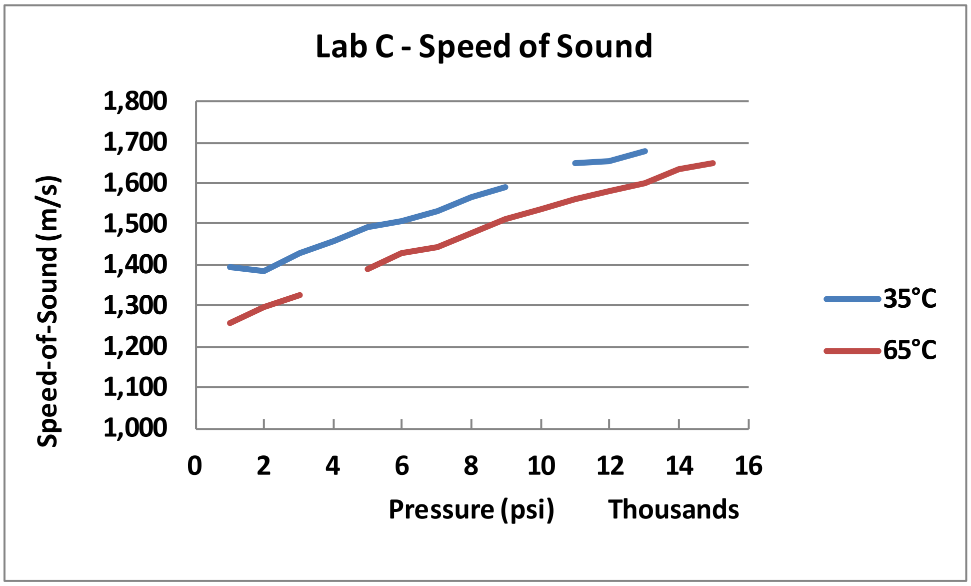


Figure D-8. Sample 6485, Lab C, Speed-of-Sound

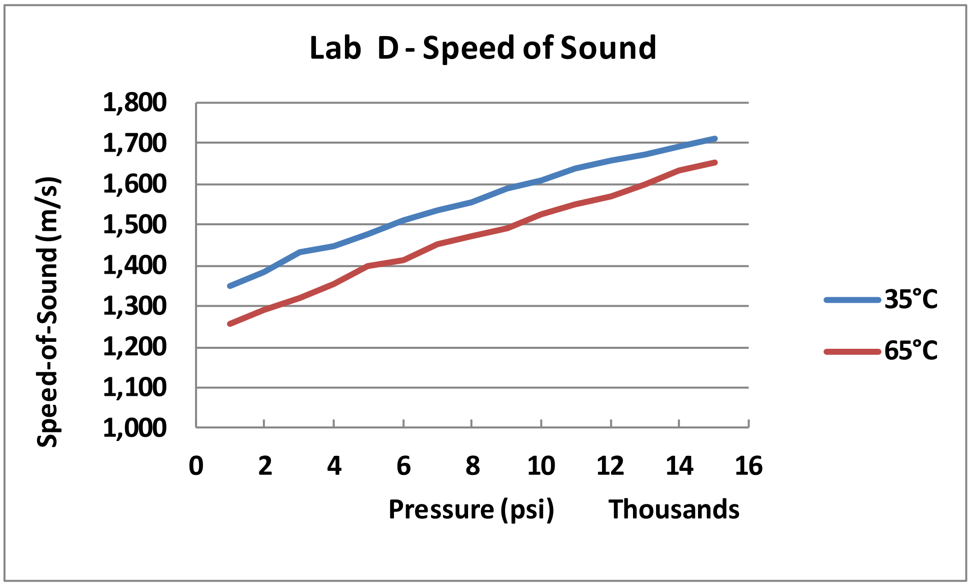


Figure D-9. Sample 6485, Lab D, Speed-of-Sound

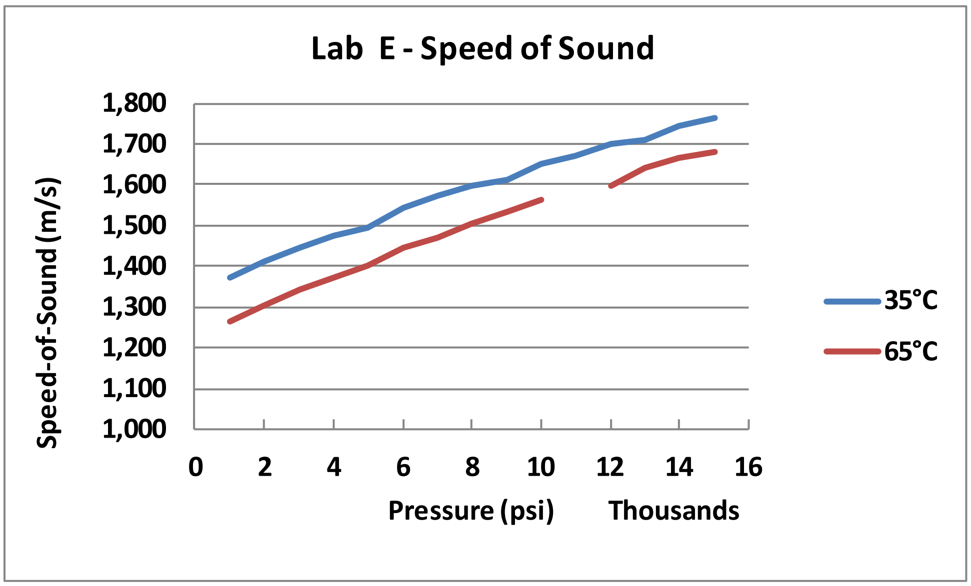


Figure D-10. Sample 6485, Lab E, Speed-of-Sound

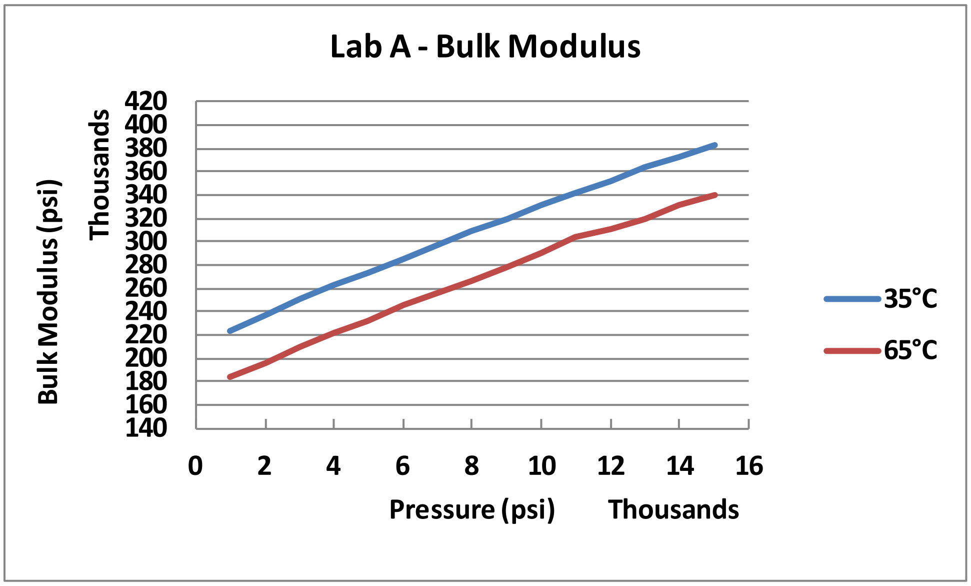


Figure D-11. Sample 6485, Lab A, Bulk Modulus

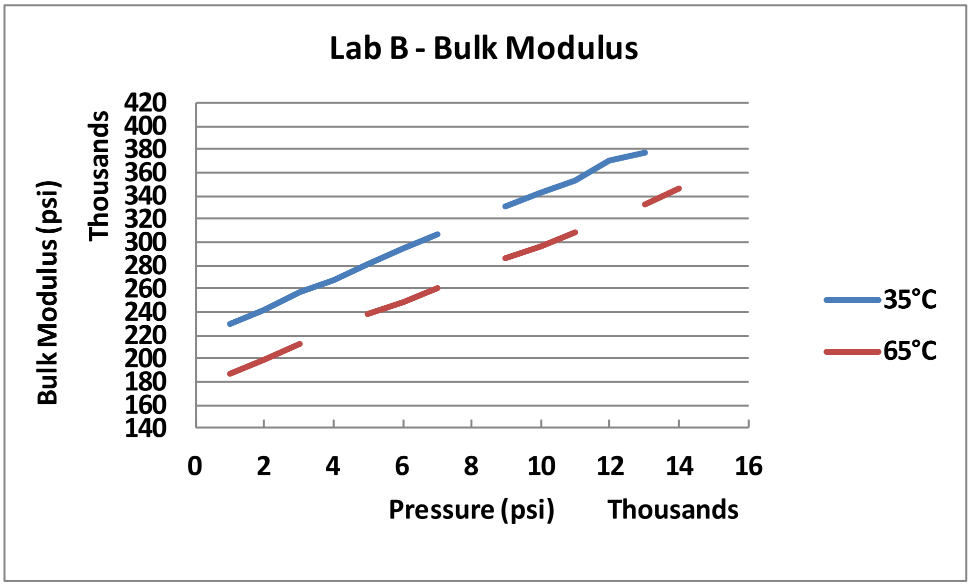


Figure D-12. Sample 6485, Lab B, Bulk Modulus

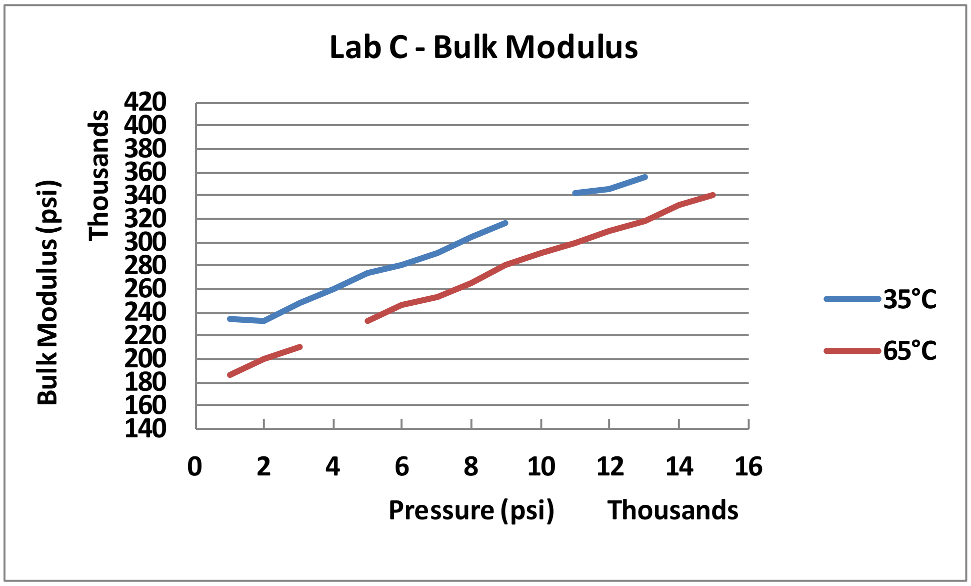


Figure D-13. Sample 6485, Lab C, Bulk Modulus

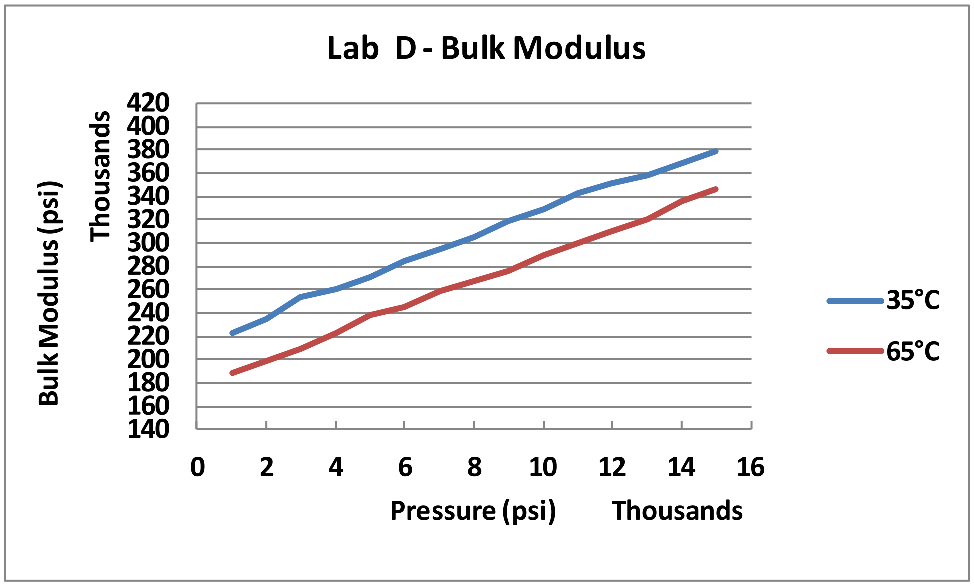


Figure D-14. Sample 6485, Lab D, Bulk Modulus

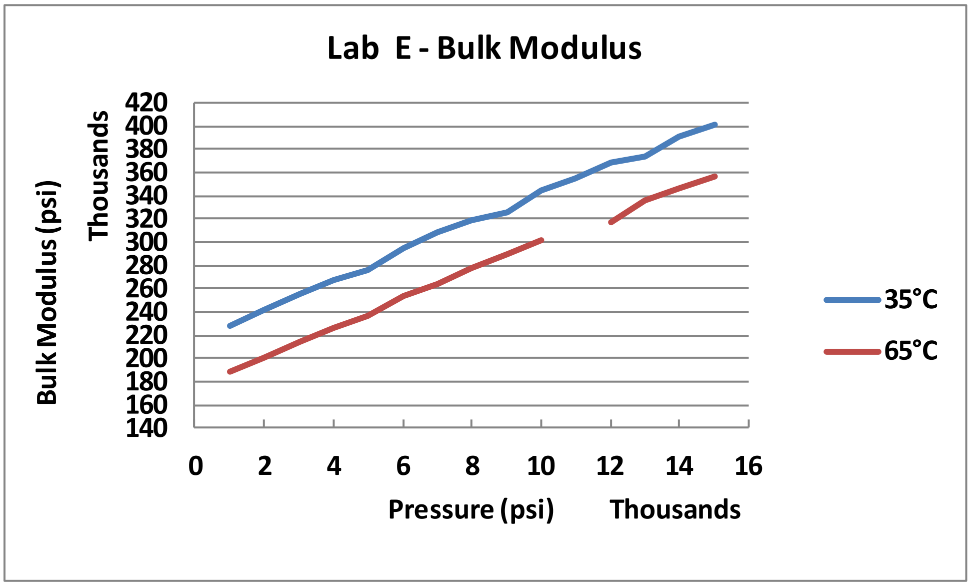


Figure D-15. Sample 6485, Lab E, Bulk Modulus

APPENDIX E.

Temperature Comparison – Sample 6486

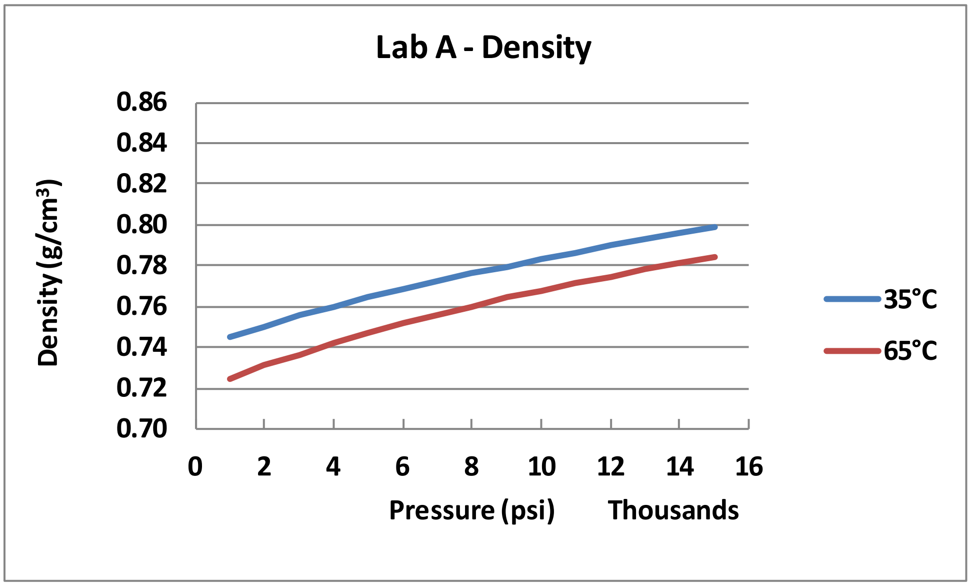


Figure E-1. Sample 6486, Lab A, Density

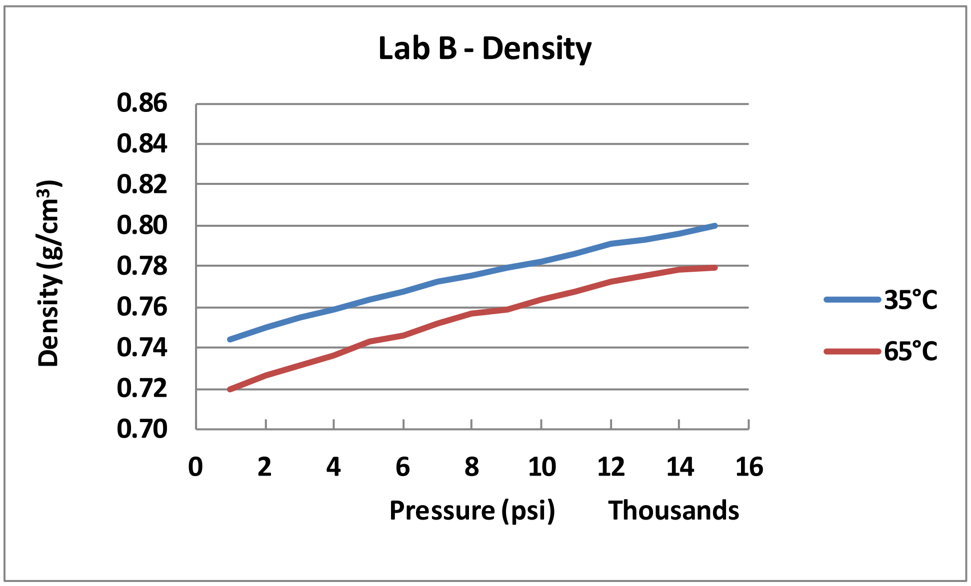


Figure E-2. Sample 6486, Lab B, Density

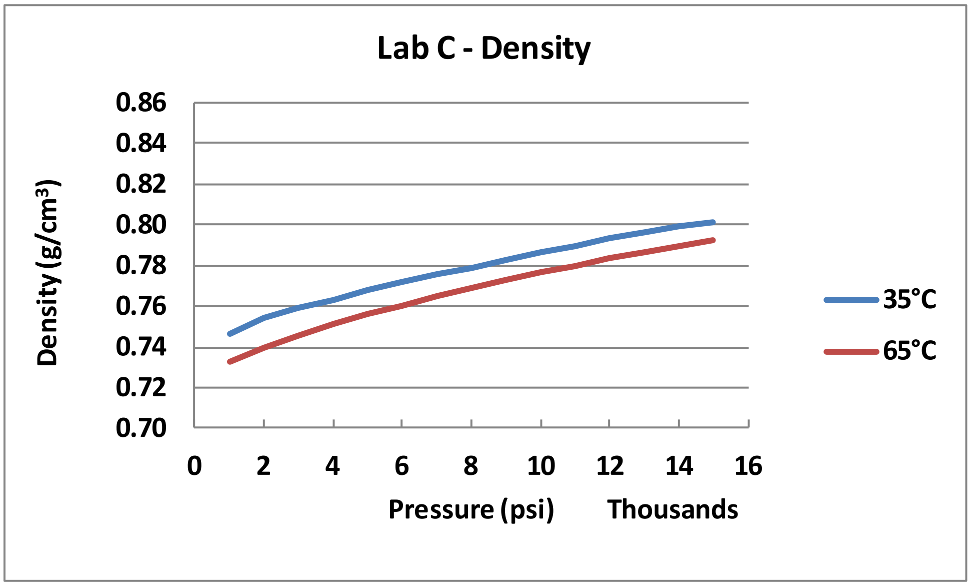


Figure E-3. Sample 6486, Lab C, Density

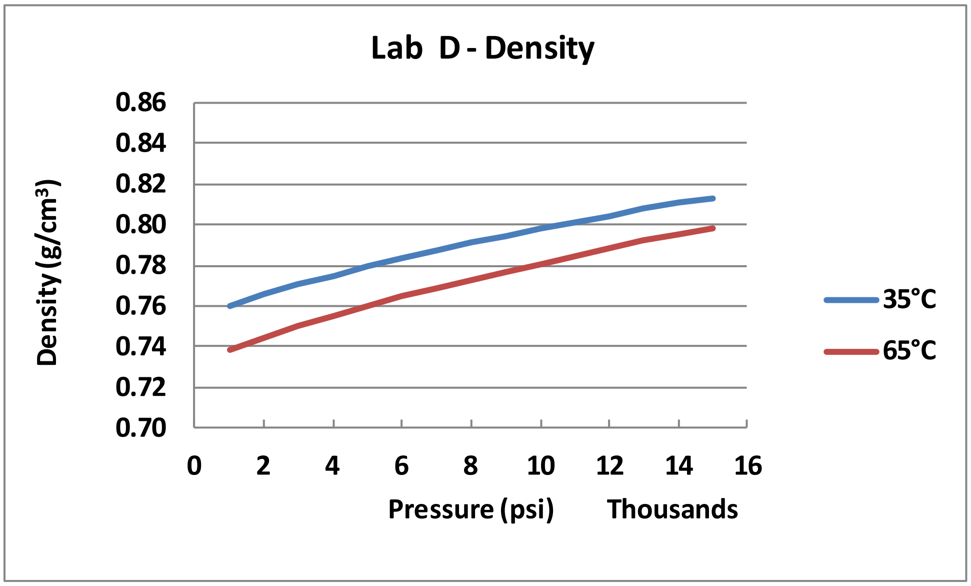


Figure E-4. Sample 6486, Lab D, Density

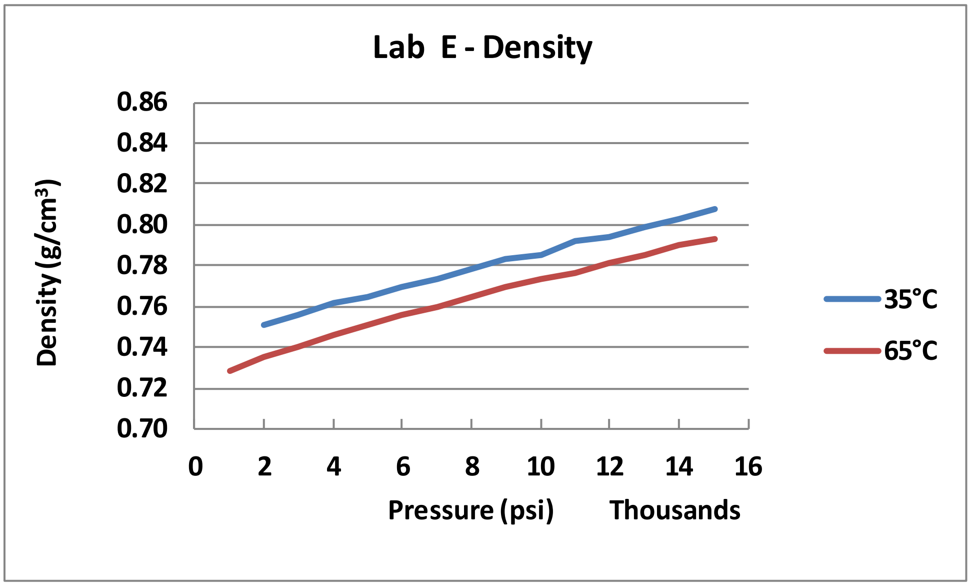


Figure E-5. Sample 6486, Lab E, Density

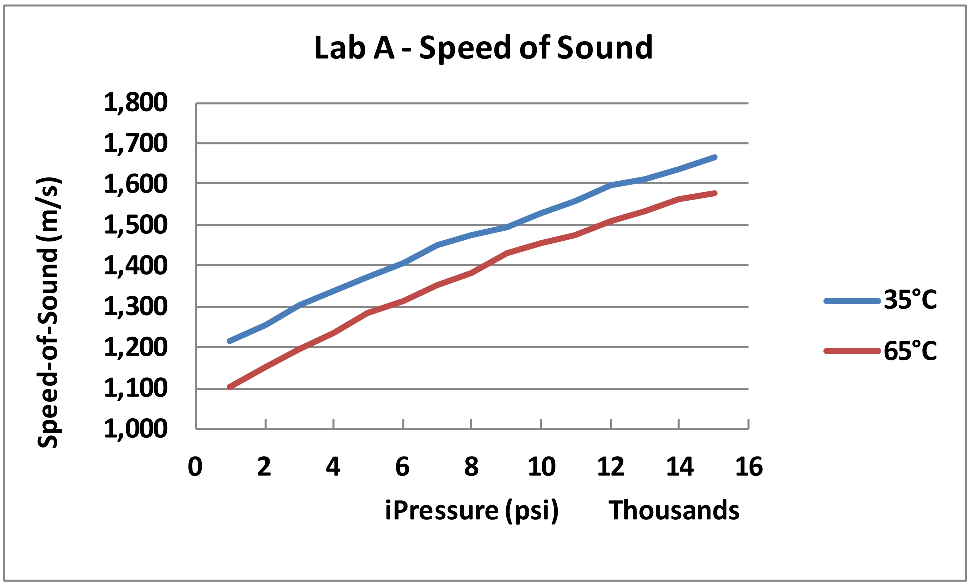


Figure E-6. Sample 6486, Lab A, Speed-of-Sound

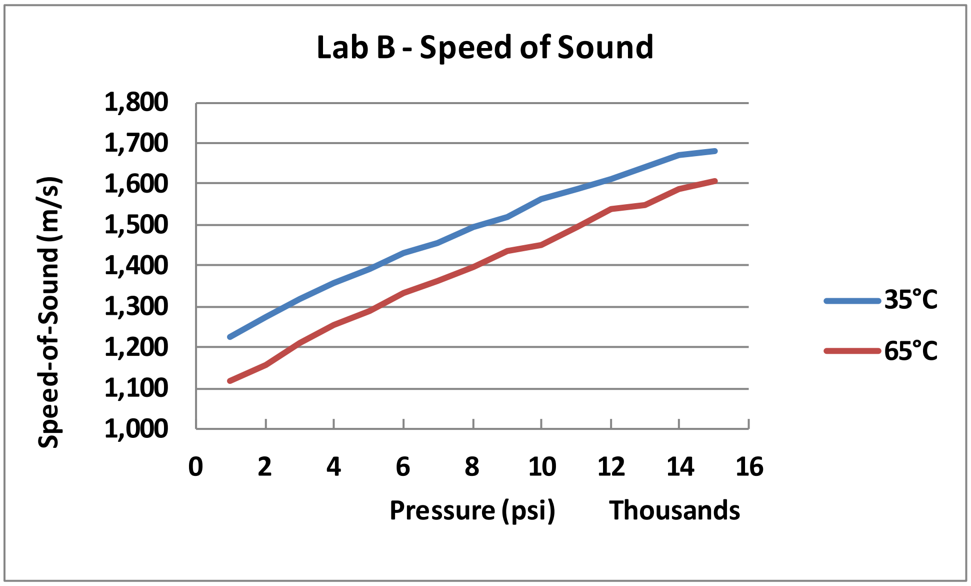


Figure E-7. Sample 6486, Lab B, Speed-of-Sound

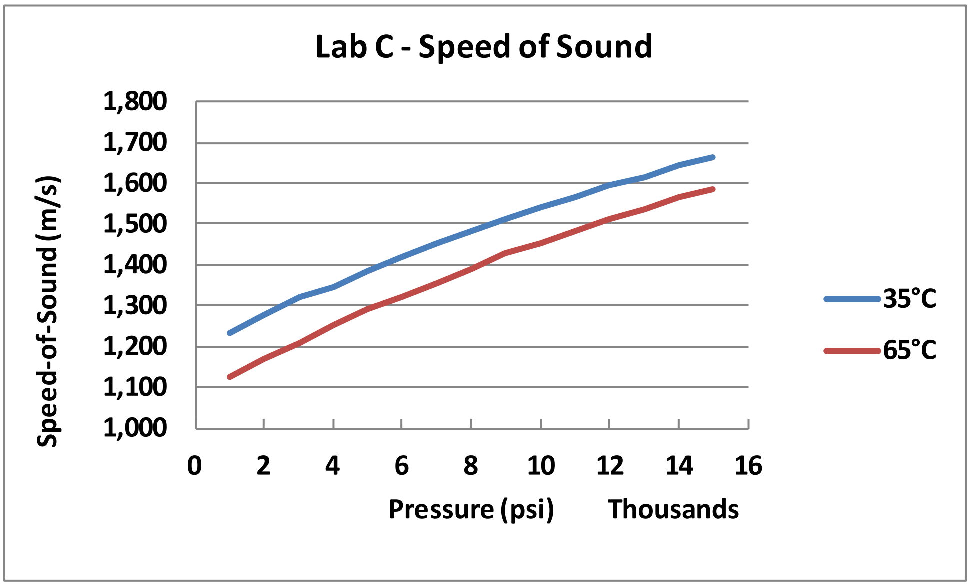


Figure E-8. Sample 6486, Lab C, Speed-of-Sound

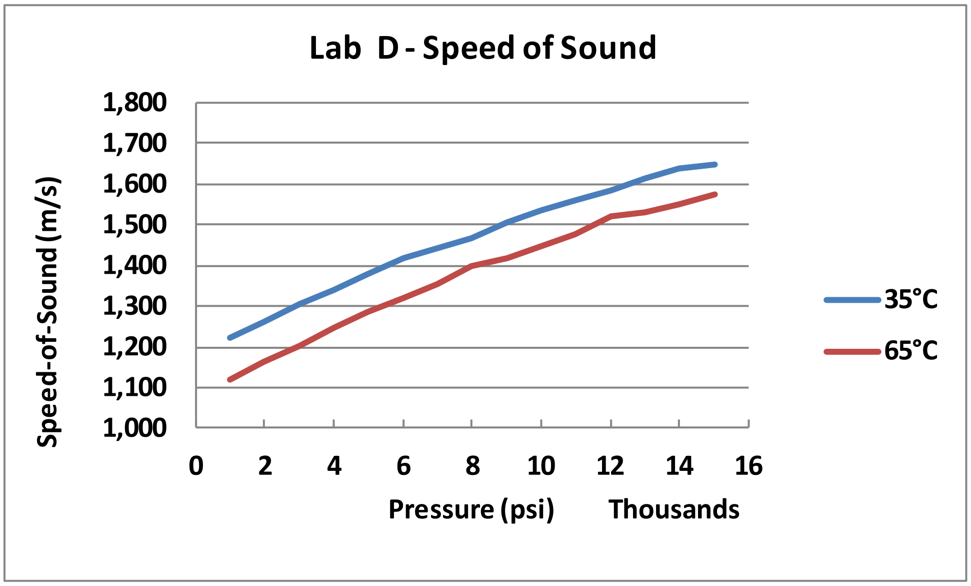


Figure E-9. Sample 6486, Lab D, Speed-of-Sound

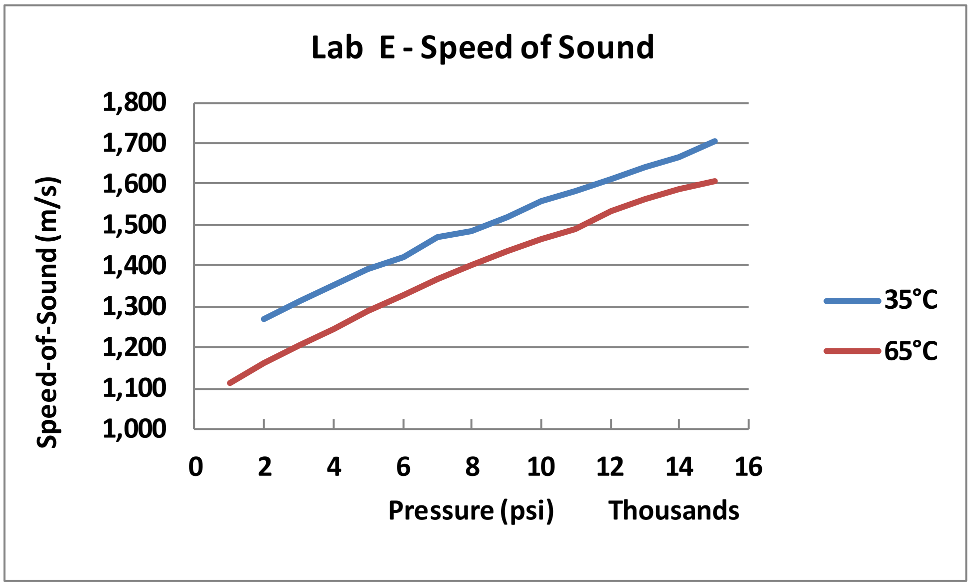


Figure E-10. Sample 6486, Lab E, Speed-of-Sound

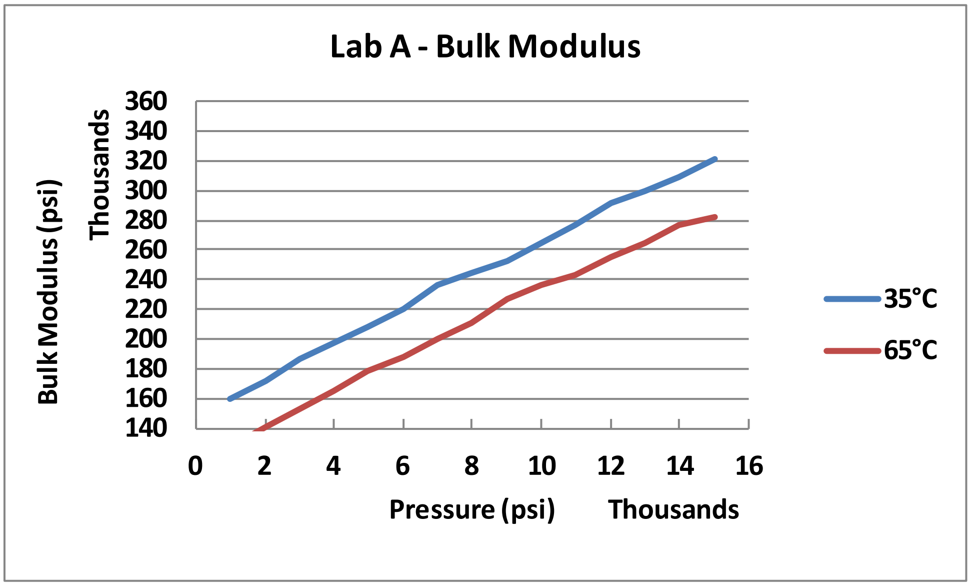


Figure E-11. Sample 6486, Lab A, Bulk Modulus

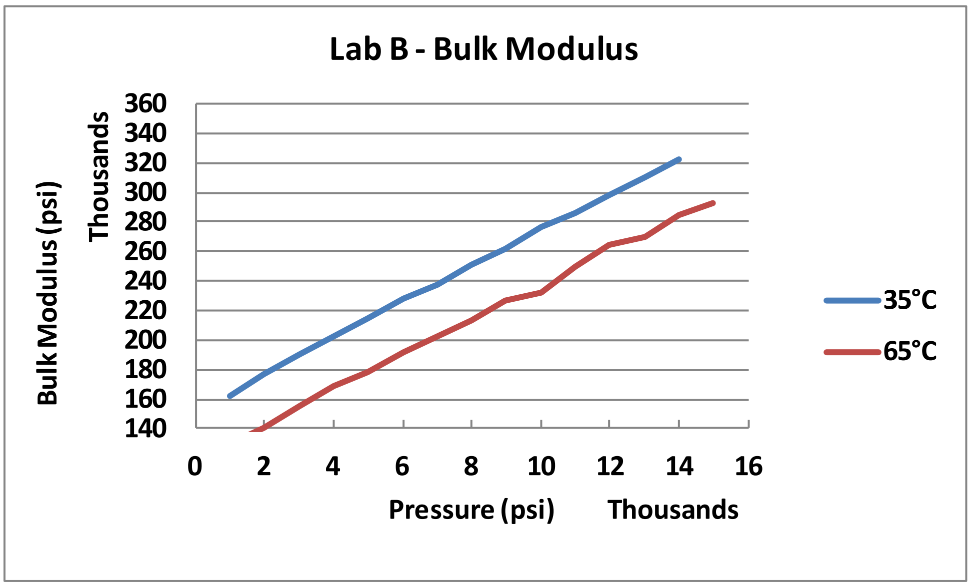


Figure E-12. Sample 6486, Lab B, Bulk Modulus

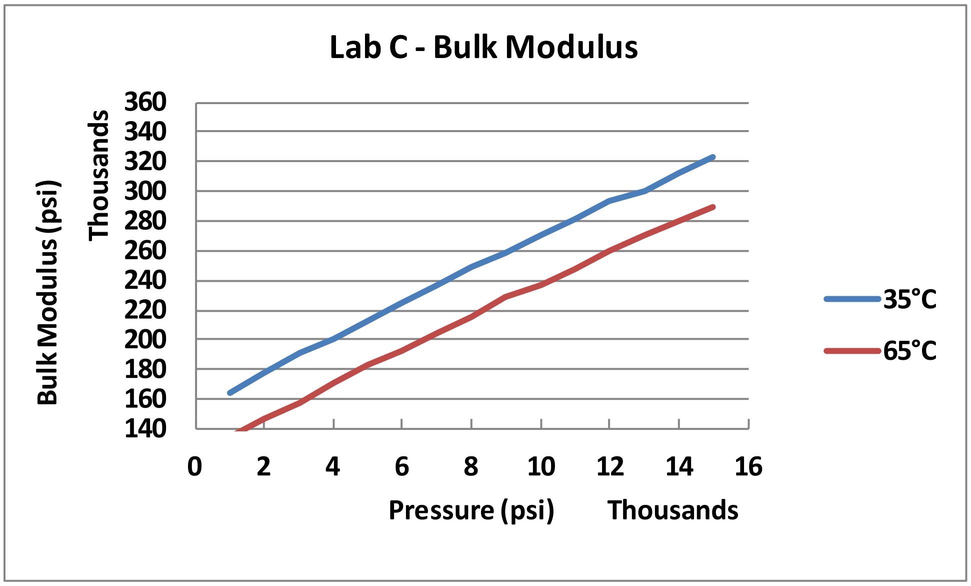


Figure E-13. Sample 6486, Lab C, Bulk Modulus

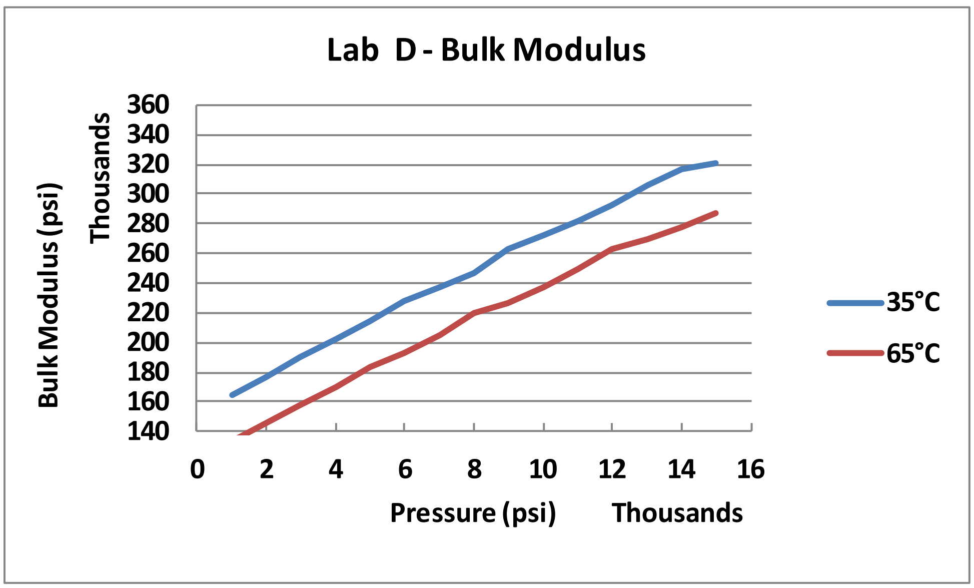


Figure E-14. Sample 6486, Lab D, Bulk Modulus

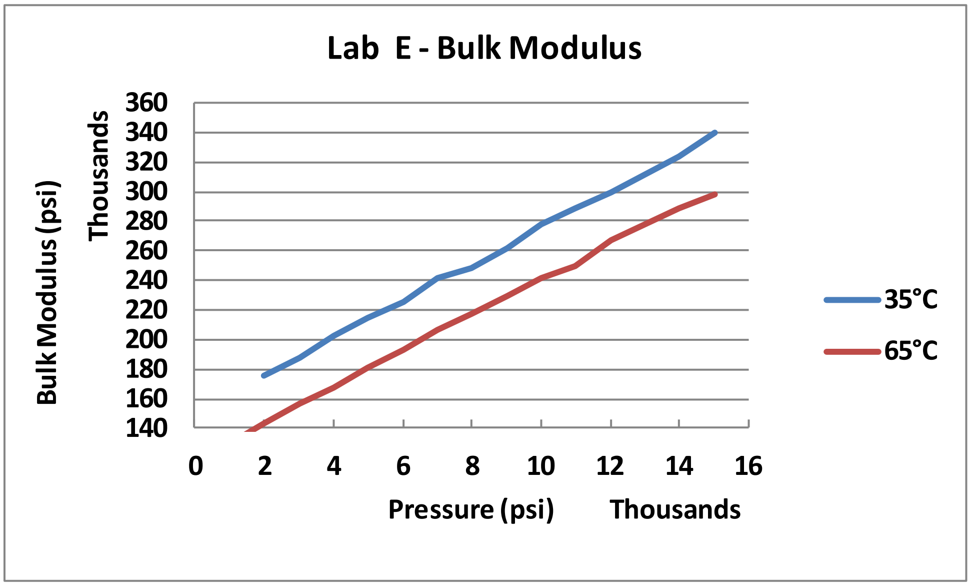


Figure E-15. Sample 6486, Lab E, Bulk Modulus

APPENDIX F.

Temperature Comparison – Sample 6487

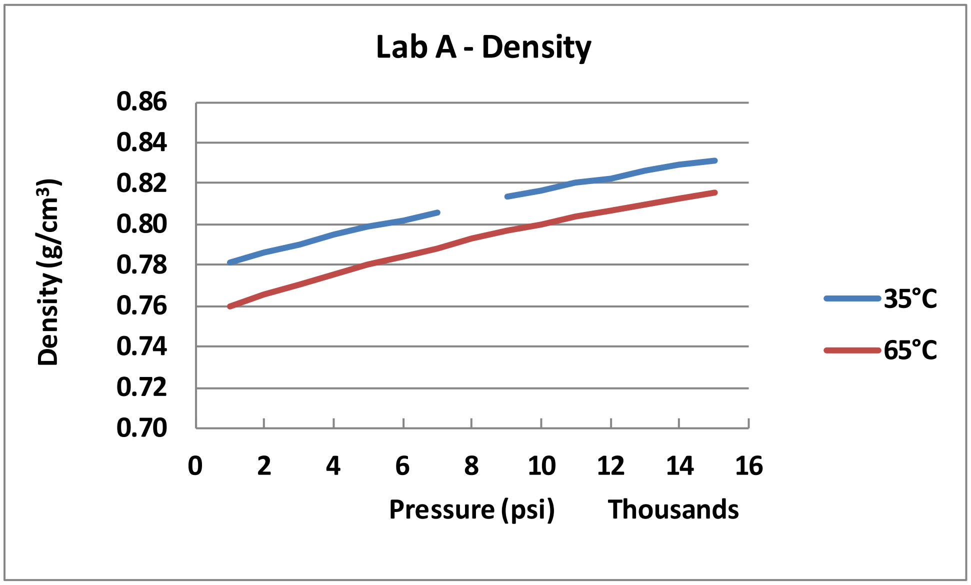


Figure F-1. Sample 6487, Lab A, Density

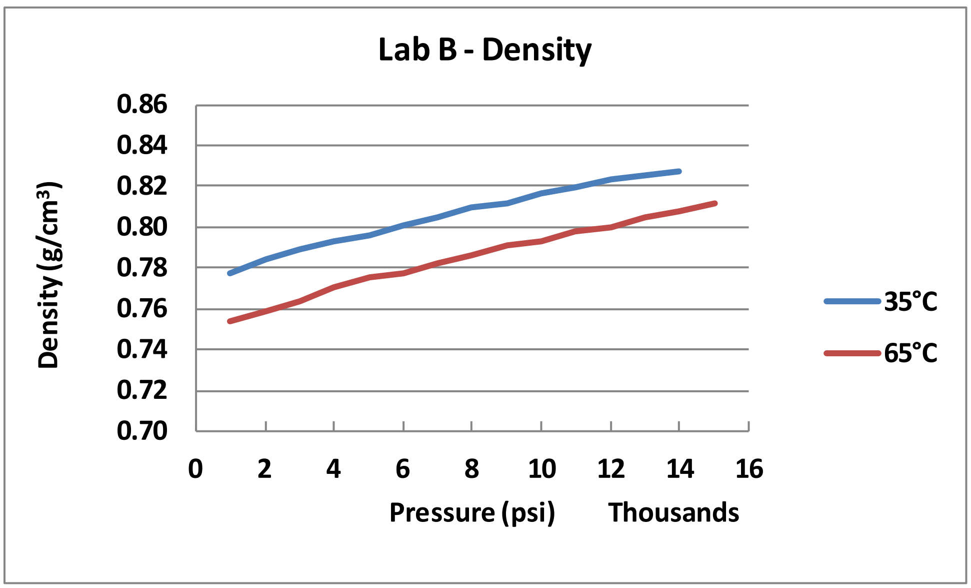


Figure F-2. Sample 6487, Lab B, Density

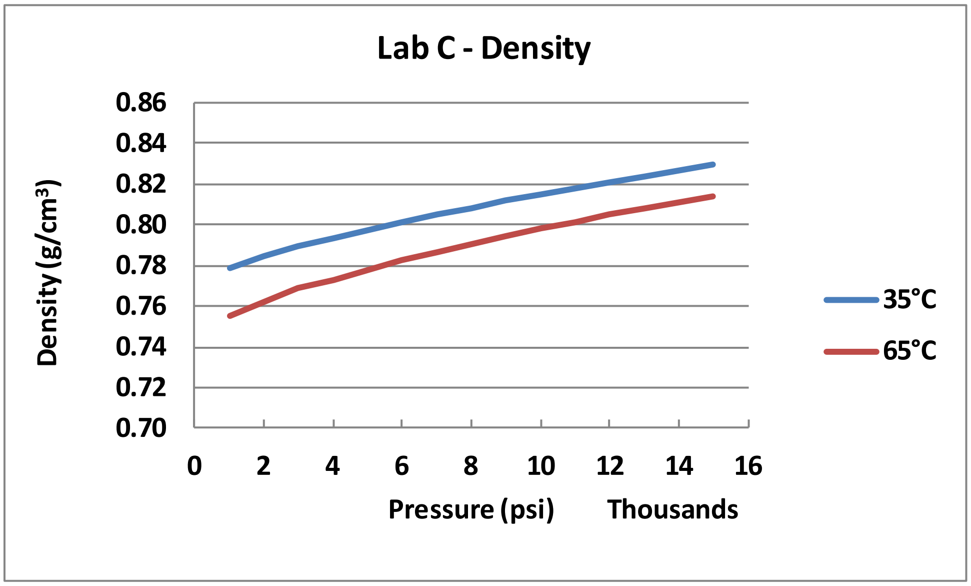


Figure F-3. Sample 6487, Lab C, Density

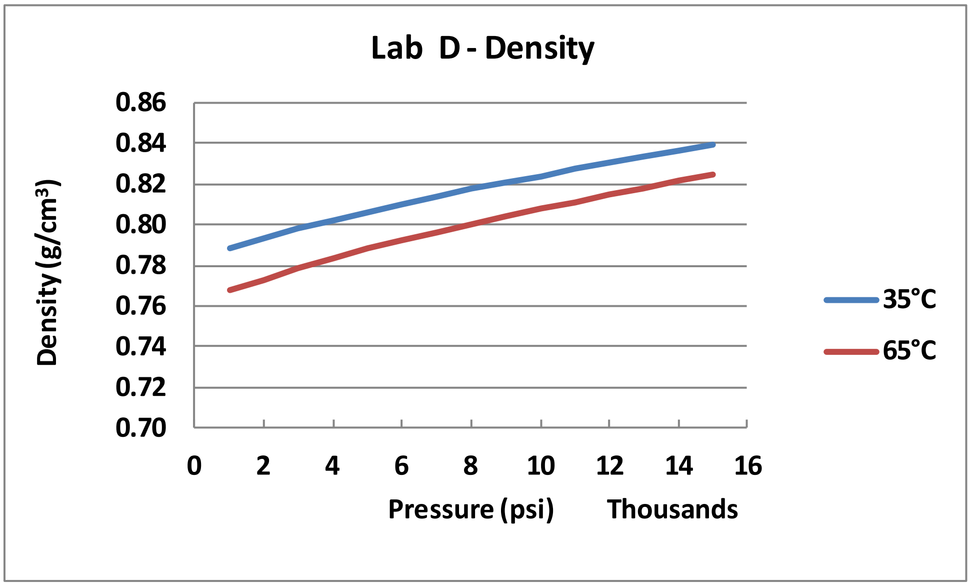


Figure F-4. Sample 6487, Lab D, Density

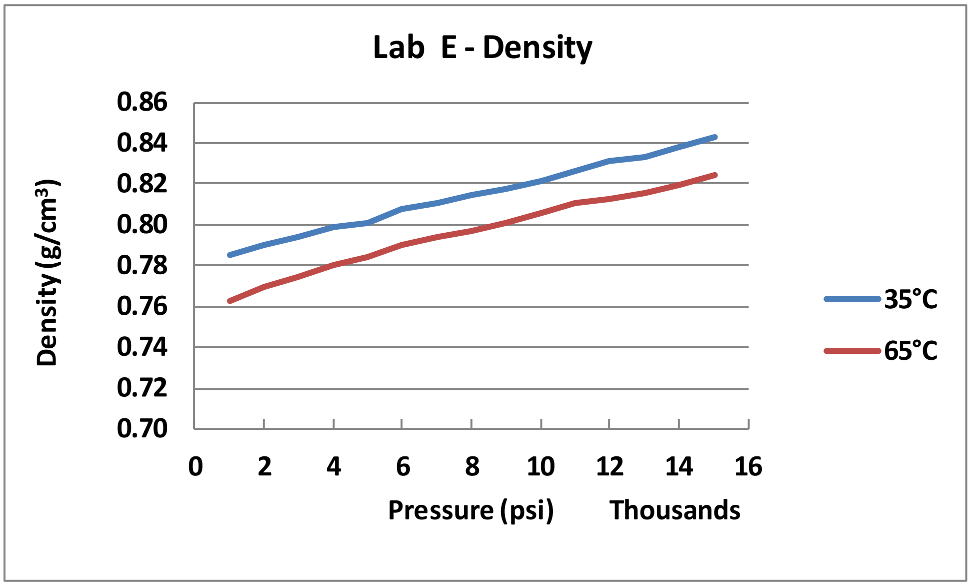


Figure F-5. Sample 6487, Lab E, Density

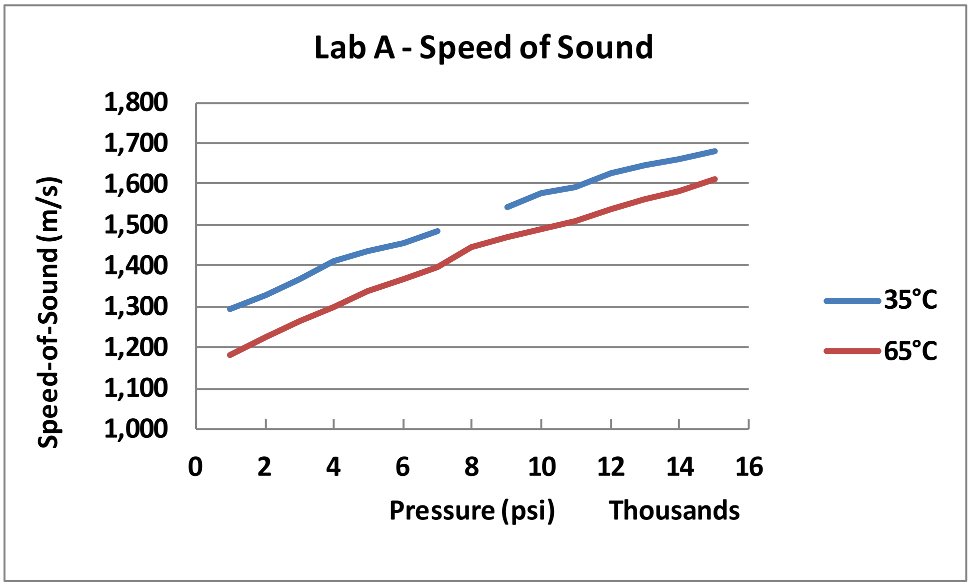


Figure F-6. Sample 6487, Lab A, Speed-of-Sound

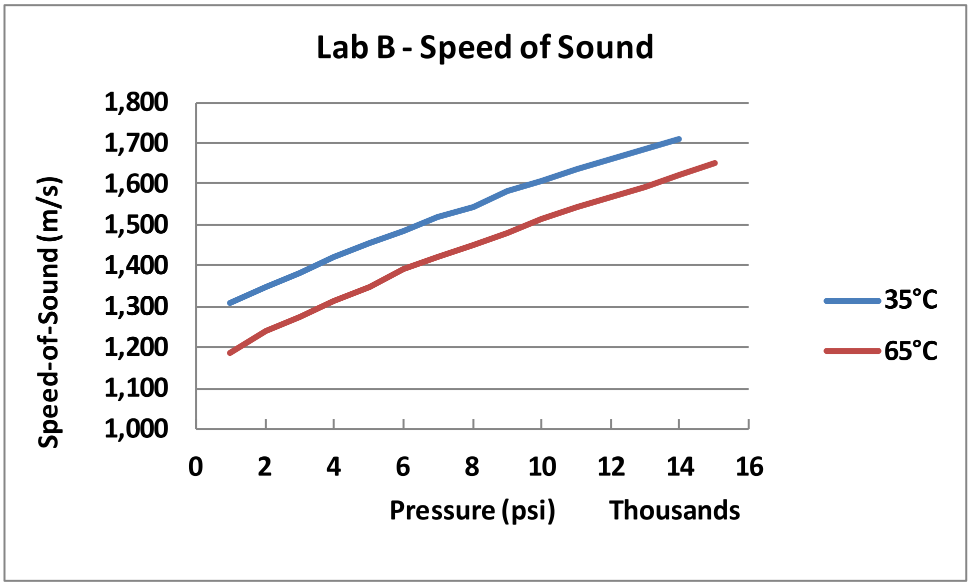


Figure F-7. Sample 6487, Lab B, Speed-of-Sound

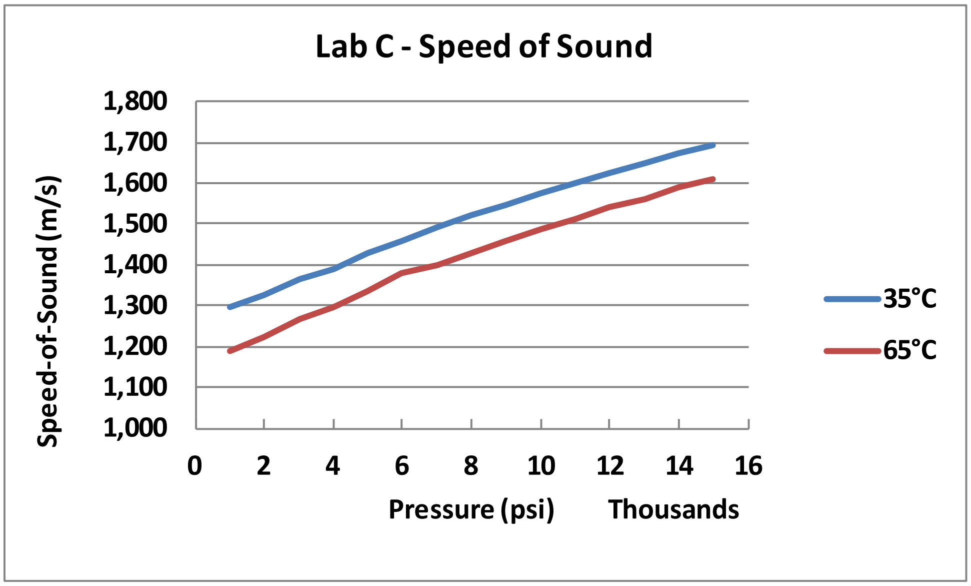


Figure F-8. Sample 6487, Lab C, Speed-of-Sound

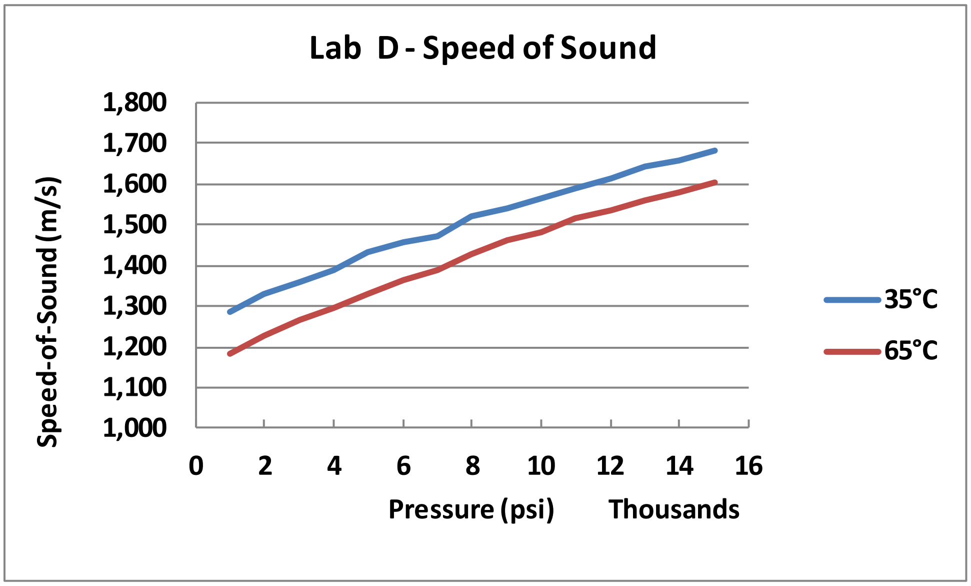


Figure F-9. Sample 6487, Lab D, Speed-of-Sound

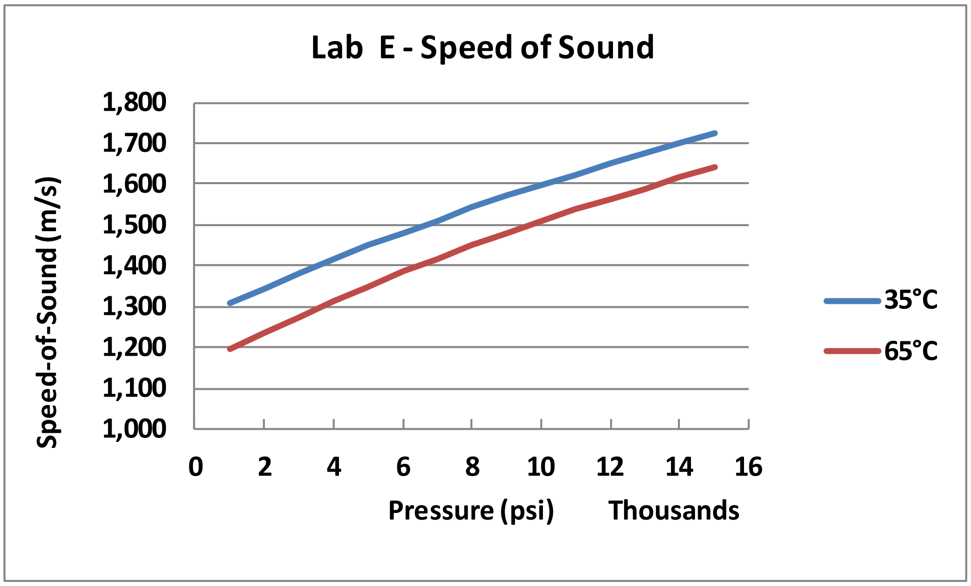


Figure F-10. Sample 6487, Lab E, Speed-of-Sound

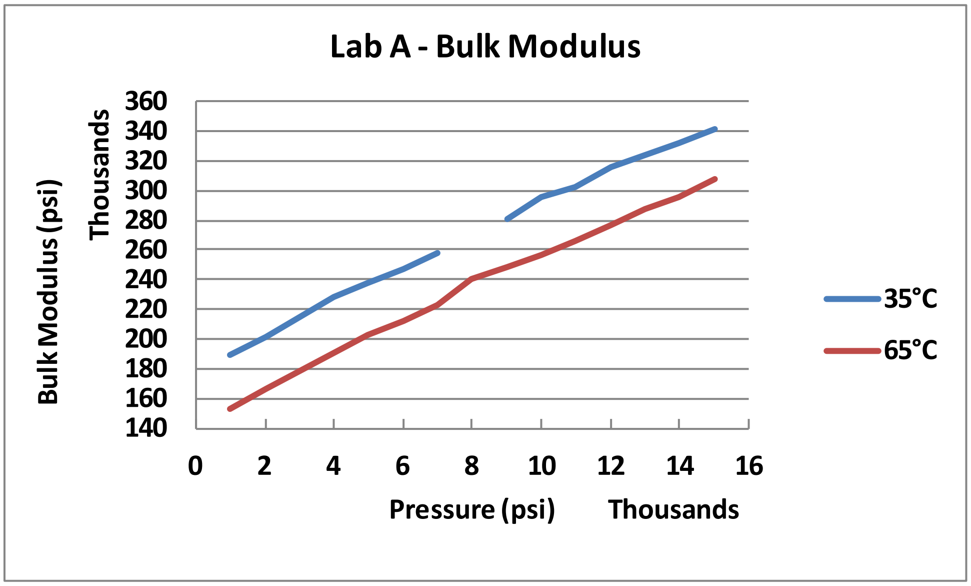


Figure F-11. Sample 6487, Lab A, Bulk Modulus

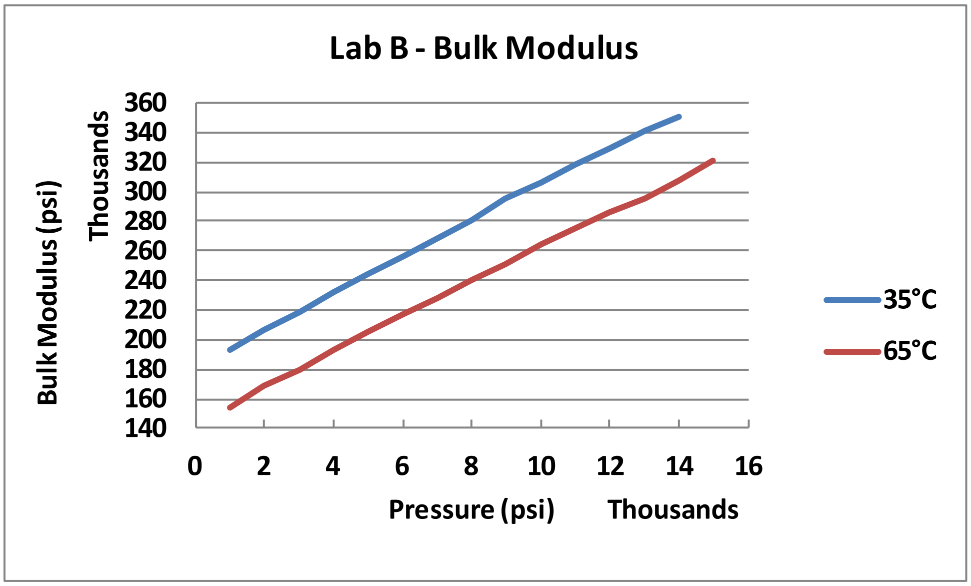


Figure F-12. Sample 6487, Lab B, Bulk Modulus

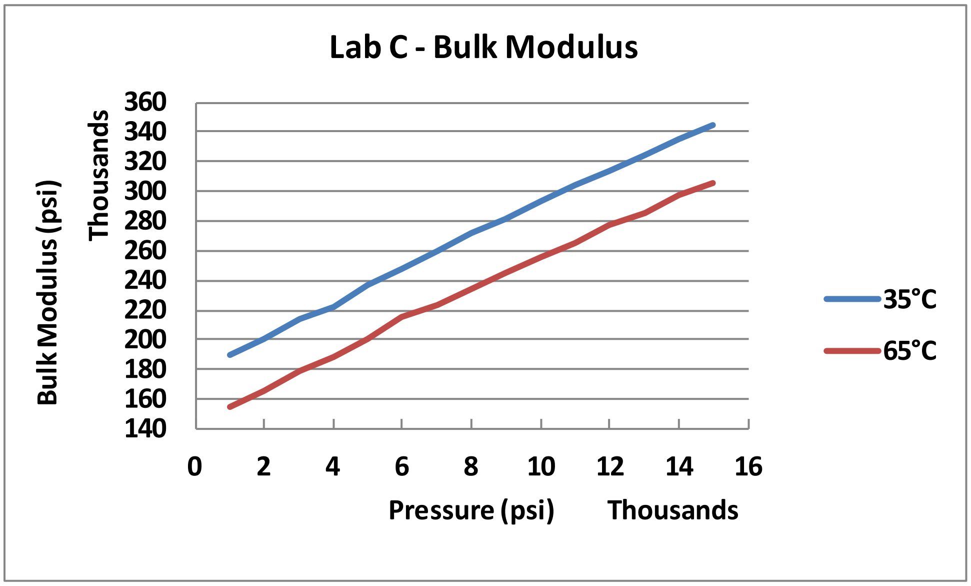


Figure F-13. Sample 6487, Lab C, Bulk Modulus

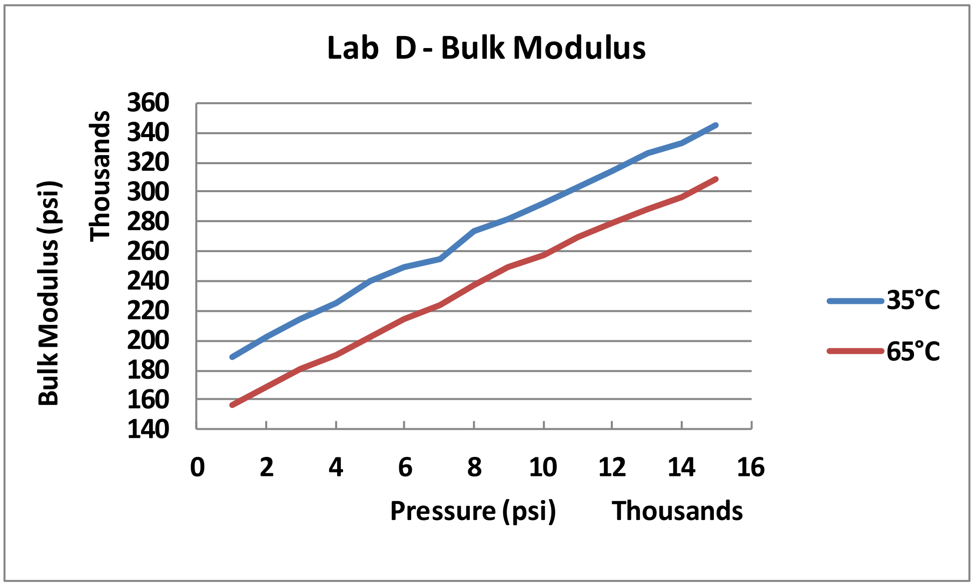


Figure F-14. Sample 6487, Lab D, Bulk Modulus

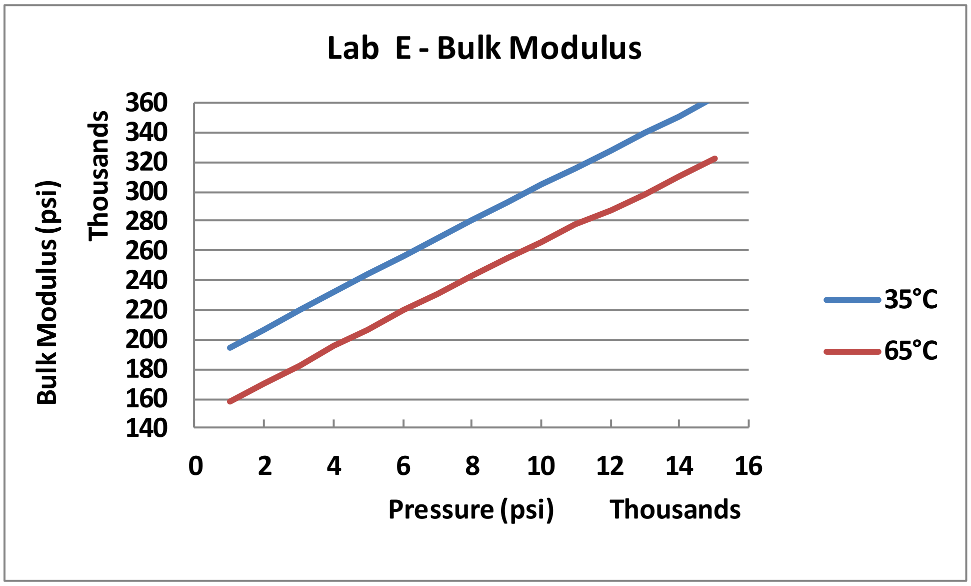


Figure F-15. Sample 6487, Lab E, Bulk Modulus

APPENDIX G.

Draft Federal Test Method

Method XXXX.X
April 1, 2015

ISENTROPIC BULK MODULUS OF AVIATION TURBINE FUELS AND DIESEL FUELS

1. SCOPE

1.1 This method describes the measurement of isentropic bulk modulus via speed-of-sound and density at temperatures ranging from 30-80 °C and applied pressures of 1000-18,000 psi. This method has been applied successfully to aviation turbine fuels and diesel fuels composed of petroleum, synthetic, and alternative feedstocks.

2. SUMMARY

2.1 This method utilizes a custom built apparatus that simultaneously measures speed-of-sound via a pulse/echo technique and density via a high temperature/pressure process densitometer. The test fuel is pumped into the system, purged of air, and sealed in a closed loop. An oven is used to equilibrate the fuel to the desired test temperature and then a pressure curve is generated by slowing reducing the volume of the system using a pressure generator. An oscilloscope is used to measure the time-of-flight of the acoustic signals generated by an acoustic transducer. Density is simultaneously measured and the bulk modulus is computed.

3. SAMPLE SIZE

3.1 The minimum recommended sample size is 500 mL.

4. STANDARDS AND APPARATUS

4.1 CAUTION – New operators should review the operating manual thoroughly before use of this equipment. This system operates at high pressures and temperatures on combustible samples.

4.2 Isentropic Bulk Modulus Apparatus consisting of the following components

4.2.1 Oven – range of 30-100 °C, stable within 1 °C

4.2.2 Data Acquisition System (DAQ) – for recording pressure, volume, temperature, and density data

4.2.3 Bulk Modulus Excel Toolkit

4.2.4 Digital Oscilloscope – for measuring pulse echoes

4.2.5 High Pressure Generator (rated for 60K psi)

4.2.6 Flow loop and valving (rated for 60K psi)

4.2.7 High Pressure Cell (rated for 30K psi max)

4.2.8 Pressure Transducer (50K psi max)

4.2.9 Pulser/Receiver Module – to drive the acoustic transducer

4.2.10 Acoustic transducer

4.2.11 Process Densitometer (rated for 20K psi at 100 °C max)

4.2.12 Densitometer Control/Display

4.2.13 Resistive Temperature Device (RTD)

4.2.14 Two (2) 500 mL beakers or sample bottles for sampling and waste

4.2.15 Secondary containment for beakers/sample bottles

4.2.16 Peristaltic pump and fuel compatible hose – for pumping fluid into the system

4.2.17 Sonicator – optional, if de-gassing sample prior to testing is preferred

5. MATERIALS

5.1 Heptanes, technical grade – for flushing the system

5.2 n-Pentane, HPLC grade – for calibration/validation of density and speed-of-sound

5.3 Isopropyl Alcohol, Certified ACS Plus - for flushing the system after the use of water

5.4 Water, deionized, ultra-filtered (DIUF) – for calibration of the densitometer

5.5 Nitrogen, dry – for leak checking or purging and drying the system (typically following an isopropyl alcohol rinse)

6. PROCEDURE

6.1 Calibration

6.1.1 System Calibration/Validation – The bulk modulus apparatus is generally calibrated/validated as a complete system. The densitometer is calibrated using n-pentane and water at multiple temperatures and pressures according to the operating manual. The speed-of-sound is calibrated using n-pentane. Overall, the system is validated by measuring the speed-of-sound and density of n-pentane at multiple pressures and a fixed temperature and plotting the results against the expected reference values. Most of the components are not user serviceable and must therefore be returned to the manufacturer for major repairs.

6.1.2 Components

6.1.2.1 Oven – Once every six (6) months, compare the oven output to a calibrated secondary reference at several temperatures

6.1.2.2 Data Acquisition System (DAQ) – adjust engineering units as needed following individual calibration of pressure and temperature components

6.1.2.3 Digital Oscilloscope – per the manufacturer's recommendations

6.1.2.4 Pressure Transducer (50K psi minimum) - Once every twelve (12) months, it is recommended that the transducer be removed and verified/calibrated by a qualified calibration lab.

6.1.2.5 Pulser/Receiver Module – per the manufacturer's recommendations

6.1.2.6 Process Densitometer - per the manufacturer's recommendations

6.1.2.7 Densitometer Control/Display - per the manufacturer's recommendations

6.1.2.8 Resistive Temperature Device (RTD) - Once every twelve (12) months, it is recommended that the RTD be removed and verified by a qualified calibration lab.

6.2 System Operation

6.2.1 Turn on the main instrument cabinet and allow the density display to initialize.

6.2.2 Turn on the oscilloscope and allow it to initialize.

6.2.3 Start the data acquisition process on the oscilloscope.

6.2.4 Load the sample into the instrument.

6.2.4.1 Ensure that the system is at atmospheric pressure.

6.2.4.2 Open all six valves (Figure 1).

6.2.4.3 Collect approximately 250 mL of sample in a beaker or other suitable container.

6.2.4.4 With the oven turned off and the door open, connect the tubing from the peristaltic pump to the inlet fitting. The other end of the tubing connected to the peristaltic pump will contain a stainless siphon tube. Place this in the beaker of fuel.

6.2.4.5 Connect another piece of tubing to the outlet fitting. If purging the system is desired, then place the stainless outlet tube into a waste container; otherwise, place the stainless outlet tube into the same beaker of fuel for recirculation. The following assumes that a purge of the previous sample with the next sample is necessary so the outlet tube should be placed in a waste container.

6.2.4.6 Turn on the peristaltic pump at full speed and ensure that sample is flowing in the proper direction toward the inlet valve (note the pump is reversible).

6.2.4.7 Close Valve C to force sample through the densitometer leg and wait for sample to exit the other end.

6.2.4.8 Once there is sample coming out of this leg, let the pump run for a few seconds to purge out the previous sample.

6.2.4.9 Close Valve A and then immediately open Valve C. This will now purge the other leg of the system.

6.2.4.10 Once there is sample coming out of this leg, let the pump run for a few seconds to purge out the previous sample.

6.2.4.11 Repeat these steps several times, alternating between one leg and the other always making sure to close one valve before opening the other.

6.2.4.12 Open Valve A and Valve C allowing sample to flow through both paths.

6.2.4.13 Cycle the high pressure generator slowly (~1 full turn every 4-5 seconds) until the piston is fully retracted (rising stem extended rearward) and then cycle the piston again to the forward position. This will flush out the piston chamber. Repeat once.

6.2.4.14 The system should now be adequately flushed. Place the stainless outlet tube into the 250 mL beaker containing the sample. The sample will now recirculate. Add additional sample to the beaker as needed if additional flushing is warranted.

6.2.4.15 At this point, additional purging/flushing can be achieved by alternate flushing of the individual legs and/or cycling of the pressure generator as described above. The key is to ensure that no air bubbles can be seen exiting the outlet fitting.

6.2.4.16 When ready to continue, slowly cycle the pressure generator until the piston is approximately 3/4 retracted (rising stem extended).

6.2.4.17 Once all of these steps have been carried out and no air bubbles are seen exiting the system, close the outlet valve, then immediately close the inlet valve, and turn off the peristaltic pump in that order.

6.2.4.18 To test whether the sample fill and air purge was successful, very slowly cycle the piston forward (clock-wise) while monitoring the pressure on the mPDS-5 display. If the system is sufficiently packed, then an immediate increase in pressure will be observed (having turned the pressure generator wheel only a few degrees). If not, then additional purging is required (re-open the inlet and outlet valves, turn on the peristaltic pump, and continue the purge process in recirculation mode).

6.2.4.19 If satisfied that the system is ready to run, with the inlet and outlet valves closed, disconnect the tubing from the inlet and outlet valves and place a Swagelok cap on each fitting.

6.2.4.20 Close the oven door.

6.2.5 Turn on the oven, set the temperature, and allow the system to equilibrate (~ hours).

6.2.6 Turn on the Pulser/Receiver.

6.2.7 Open a copy of the Bulk Modulus Excel Toolkit and begin acquiring data.

6.2.8 Increase the system pressure to the target value and allow to stabilize (~ minute(s)).

6.2.9 Measure the time-of-flight of the acoustic pulse on the oscilloscope and record the data into the Excel Toolkit.

6.2.10 Repeat steps 8-9 for each target pressure.

6.2.11 When complete, return the system pressure to baseline conditions (~atmospheric).

6.2.12 Turn off the Pulser/Receiver (when not in use)

6.2.13 If desired, change the oven temperature to the next target value, allow to equilibrate, and repeat steps 6-12.

6.2.14 Otherwise, set the oven temperature to ~25 °C and allow the system to cool (~ hour(s))

6.2.15 Flush the system

6.2.16 Exit out of all software and power down the oscilloscope.

6.2.17 Turn off the main instrument cabinet.

7. CALCULATIONS

7.1 Isentropic Bulk Modulus is calculated as $\beta=\rho c^2$, where β is the bulk modulus in Pa, ρ is the density in kg/m^3, and c is the speed-of-sound in m/s.

8. REPORTING

8.1 For a given temperature and pressure, report the following:

8.1.1 Test Temperature in °C to the nearest 0.1 °C

8.1.2 Isentropic Bulk Modulus in psi to the nearest integral value

8.1.3 Speed-of-Sound in m/s to the nearest 0.1 m/s

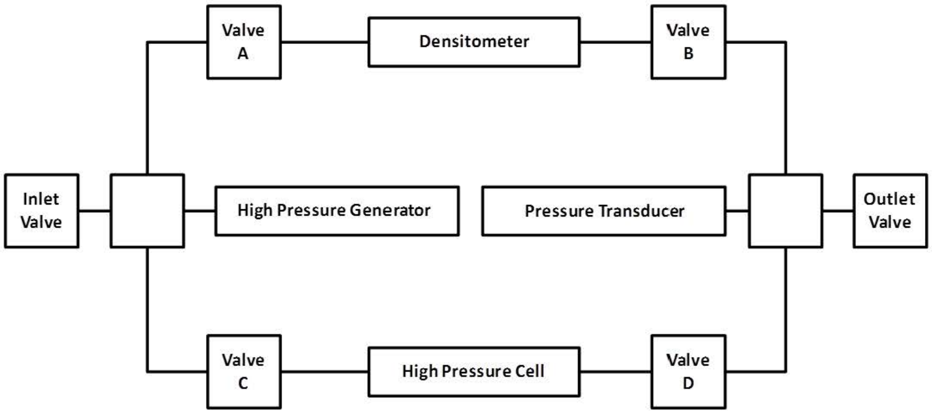
8.1.4 Density in kg/m^3 to the nearest 0.1 kg/m^3

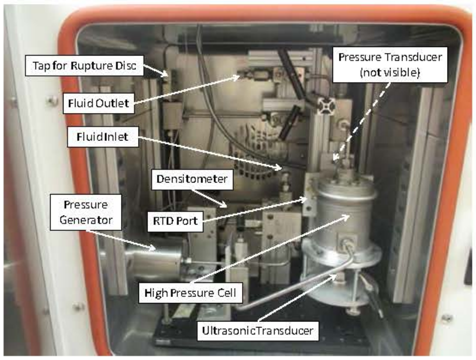
9. PRECISION

9.1 Repeatability – pending final computation

9.2 Reproducibility – pending final computation

Method Prepared by:
XXXX – XXXX - 2014





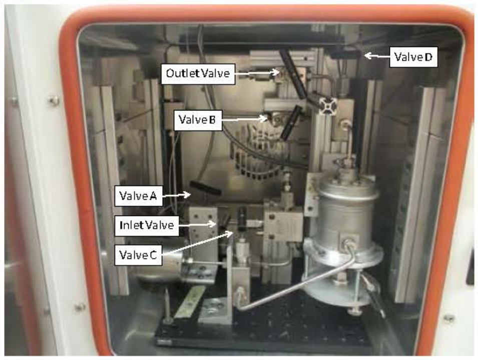


Figure 1. Instrument Flow Loop Schematic